

MASS-TRANSFER TESTS WITH TWO-PHASE FLOW

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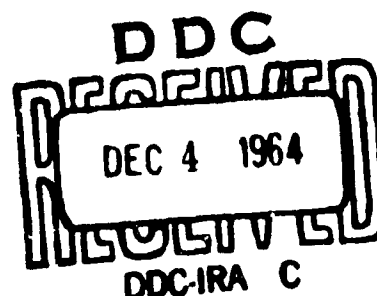
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Air Force Aero Propulsion Laboratory  
Research and Technology Division  
Air Force Systems Command  
Wright-Patterson Air Force Base, Ohio

Project No. 3145, Task No. 314511

(Prepared under Contract AF33(657)-8954  
by AiResearch Manufacturing Company of Arizona,  
a division of The Garrett Corporation, Phoenix, Arizona,  
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## FOREWORD

This report discusses the work performed by the AiResearch Manufacturing Company of Arizona, a division of The Garrett Corporation, Phoenix, Arizona, as a part of the SPUR power conversion-system studies. The work was performed under United States Air Force Contract AF33(657)-8954. This contract was initiated under Project No. 3145, "Dynamic Energy Conversion," Task No. 314511. "Nuclear Mechanical Power Unit." The contract was administered by the Research and Technology Division, AF Aero Propulsion Laboratory, Flight Vehicle Power Branch, United States Air Force Systems Command, with Mr. C. H. Armbruster as the Project Engineer.

The SPUR engineering program at AiResearch was directed by Mr. J. H. Dannan, SPUR Project Engineer, and the work reported herein was conducted under the supervision of Messrs. E. A. Kovacevich and R. L. Salley. Acknowledgment is given Messrs. H. Tom and L. Timmers of the AiResearch Laboratory for their contributions to this effort.

This report covers activities conducted at AiResearch to December 1963 on the "Mass-Transfer Tests with Two-Phase Flow."

The supplementary report number assigned to this report by The Garrett Corporation is SY-5463-R.

## ABSTRACT

A closed loop fabricated entirely from Cb + 1.0 w/o Zr material was operated with potassium under dynamic two-phase (liquid and vapor) conditions. A test section containing orifices and Mo + 0.5 w/o Ti test specimens was placed in the vapor stream. The maximum fluid temperature of 1950°F and the maximum vapor temperature of 2000°F were maintained throughout the test. Condensing occurred at 1770°F. The test was conducted for 307 hours of the scheduled 1,000 hours. The test was terminated due to a twisted ribbon failure in the boiler section, which resulted in a flow restriction. The test was conducted in a purified argon atmosphere and was monitored for impurities during the test. Despite the precautions taken to minimize contamination of the loop material, contamination of the refractory alloy existed. Under the test conditions, there was no evident corrosion and/or erosion of the loop materials.

Publication of this technical documentary report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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## SECTION I - INTRODUCTION

The use of alkali metals such as potassium as working fluids in space power systems has created recognized problems concerning materials, especially at elevated temperatures. The most evident problem is that of alkali-metal containment at the desired system operating temperatures. Many investigators have conducted liquid-compatibility tests, both dynamic and static, with refractory metals to obtain corrosion information up to 2000°F. However, dynamic alkali-metal corrosion data from two-phase (liquid and vapor) tests is sparse. These tests are limited to a few research organizations and, in most instances, are operated to yield information for a given set of test conditions which may not be directly comparable to the power-conversion system under study, namely, SPUR. For example, the SPUR boiler design employs forced vortex boiling; and in order to simulate the test conditions in a compatibility test, a tube-type boiler and not a pool-type boiler should be employed. A review of past and present alkali-metal corrosion investigations revealed that dynamic two-phase tests where forced-circulation boiling is used and temperatures are in the SPUR temperature range are nonexistent. Therefore, the purpose of this investigation was to obtain compatibility data under comparable SPUR conditions with SPUR materials utilized--namely, a columbium alloy, Cb + 1 w/o Zr, and a molybdenum alloy, Mo + 0.5 w/o Ti.

The accepted method of obtaining the compatibility data for liquid and vapor potassium under elevated temperatures and dynamic conditions is a closed-loop test. The loop used for the tests described in this report was fabricated entirely from Cb + 1.0 w/o Zr. Inserts of Mo + 0.5 w/o Ti (a candidate material for the SPUR turbine rotor) were placed in the vapor region of the Cb + 1.0 w/o Zr loop. The design test conditions for this compatibility test are summarized below.

Boiling temperature:	1950°F
Superheat temperature:	2000°F
Condensing temperature:	1500°F ±50°F
Subcooling temperature:	1100°F
Mass-flow rate:	30 lb per hr
Vapor velocity:	Mach 0.8
Maximum heat flux:	
Preheat section:	435,000 Btu per hr sq ft
Boiler section:	556,000 Btu per hr sq ft

## SECTION II - LOOP DESIGN

The loop was fabricated entirely from the columbium alloy, Cb + 1.0 w/o Zr. The loop design incorporated a pump and flowmeter, a boiler section, a vapor test section, and a condensing section. The detailed components that were employed in the loop assembly as shown in Figure 1 were as follows:

- (1) Liquid pump
- (2) Flowmeter
- (3) Throttling orifice
- (4) Heating section with turbulators or twisted ribbons
- (5) Electrical heaters
- (6) Test section containing fixed orifice plates and turbine-wheel test material (Mo + 0.5 w/o Ti)
- (7) Finned tube condenser
- (8) Surge tank with liquid-level instrumentation
- (9) Thermal insulation
- (10) Electrical insulation
- (11) Instrumentation
- (12) Argon supply

A Style 0.5-150 MSA electromagnetic pump (Serial No. 534, Catalog No. 501030) was used in the test. The pump had a rating of 28 amp, 240 volts, single phase, and 60 cycles. The pump cell was specially fabricated, since the pump tube was Cb + 1.0 w/o Zr. The nickel bus bars were brazed to the 3/8-inch-outside-diameter by 0.045-inch-wall tubing. Prior to the pump cell fabrication, various fabrication tests were operated to determine the optimum brazing alloys and procedures for joining the nickel bus bars to the Cb + 1.0 w/o Zr tubing. It was found that the brazing alloy, Coast Metals 52 (AMS 4778), gave satisfactory results. Diffusion studies on test samples brazed with this alloy were run at 1650°F, the anticipated pump operating temperature, for 200 hours. Results indicated that a 0.003- to 0.005-inch flash coating of iron on the Cb + 1.0 w/o Zr material was adequate to minimize the diffusion of nickel into the columbium-base material. Special Chromel-Alumel thermocouples were placed in key areas of the pump such as the nickel bus bars and primary and secondary coil windings.

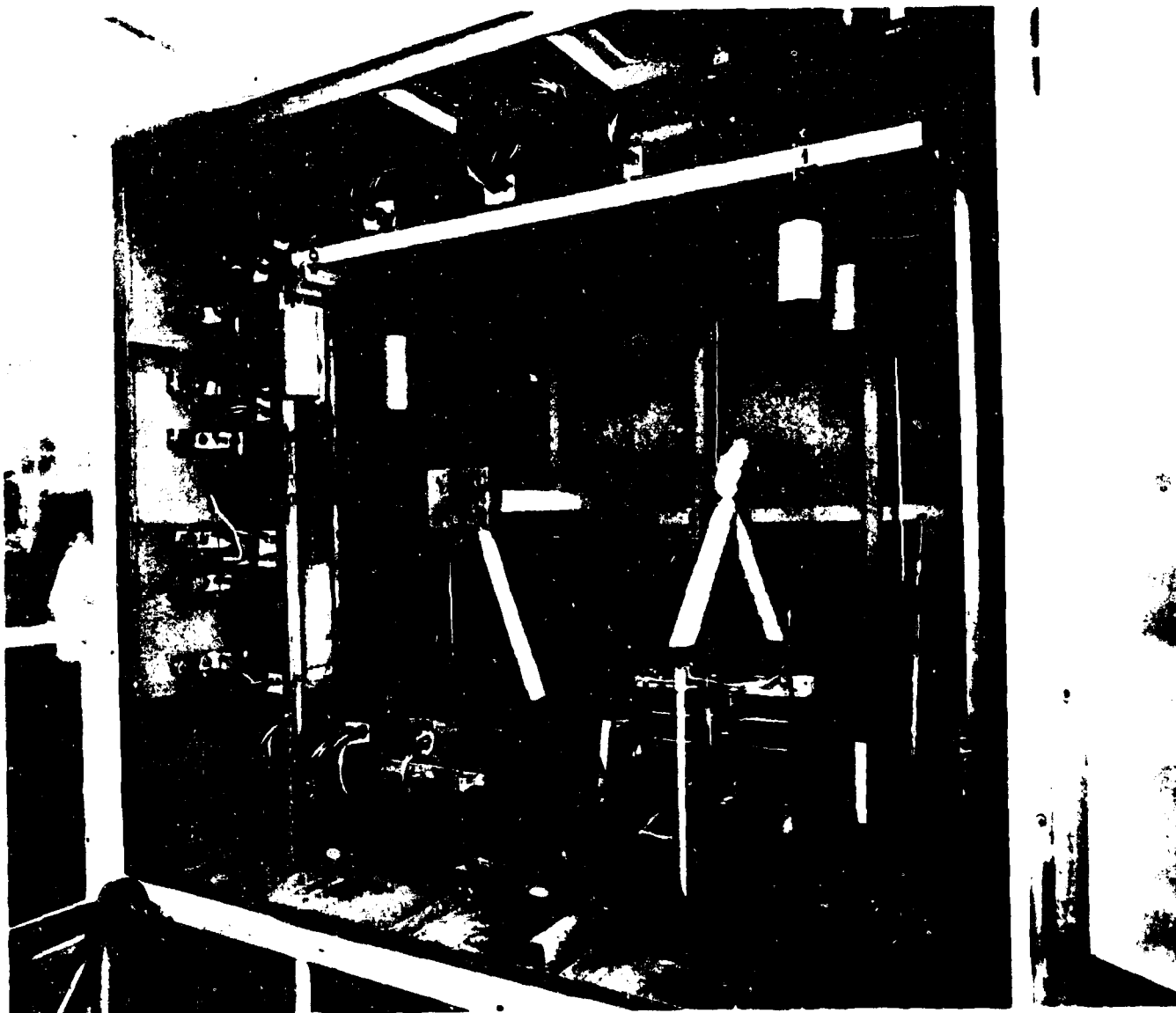


FIGURE 1. TWO-PHASE MASS-TRANSFER LOOP

For liquid flow measurement an MSA magnetic flowmeter (Serial No. 252, Catalog N . 501030) was used. This also was fabricated from 1/4-inch-outside diameter Cb + 1.0 w/o Zr tubing having a wall thickness of 0.030 inch. The permanent magnet, Serial No. 63, was measured at 3030 gauss

A liquid throttling orifice fabricated from Cb + 1.0 w/o Zr material was placed in the loop after the flowmeter and prior to the heater section. The fixed orifice, 0.030 inch in diameter, was sized for the loop operating conditions. The purpose of the orifice was to facilitate stable boiling conditions in the boiler.

The heating section consisted of Cb 1.0 w/o Zr tubing (Wah Chang, Heat No. 88-1687), 5/16 inch in outside diameter with a 0.049-inch wall, containing a twisted Cb + 1.0 w/o Zr ribbon, 0.015 inch thick (Wah Chang, Heat No. 8-2033), on the inside tube annulus. The ribbon was placed in the tube after the flowmeter and throughout the heater sections to the test section. The ribbon was twisted one complete turn per inch of length. The turbulator was a single piece of ribbon with the ends welded as part of the main loop tube weldments. The ribbon width was such that it was a slip fit inside the 5/16-inch-OD tubing. No effort was made to attach the ribbon to the inner tubing wall.

The heater section consisted of a 2-foot-long preheater section containing two 6-kw tantalum resistance heaters, a 4-foot-long boiler section containing three 8-kw tantalum heaters, and a superheat section also containing three 8-kw tantalum heaters. Each heater was individually controlled by powerstats.

The heaters were fabricated from 5/8-inch tantalum tubing (National Research Corporation, Heat No. 2144) having a wall thickness of 0.015 inch. The heaters were prefabricated as clamshells to facilitate assembly in the loop after its fabrication. Tantalum bus bars, 1/2 inch wide and 3/32 inch thick, were attached by hand brazing to the heater halves at each end of the heater. The braze material in this case was 0.001-inch-thick pure columbium foil. Brazing was conducted under inert conditions in a triple-evacuated and triple argon purged vacuum dry box. Upon attachment to the loop the heater halves were then TIG-welded together under high purity inert gas conditions.

Additional heaters for weld annealing (up to 2200°F) and adiabatic wall conditions for temperature measurement were fabricated from tantalum wire. These heaters were placed in areas where field welds were made for loop assembly and in areas where accurate thermocouple readings were required. A typical heater is shown in Figure 2. The tantalum heating wire was held in place within the tantalum outer shells by means of boron nitride insulators. The outer tantalum halves containing the heater wire were placed on the loop and then TIG welded in several areas to hold the heater together. Tantalum wire was also used in the heater assembly and was not removed after completion of the assembly. Figure 3 shows the assembled heater on the loop.

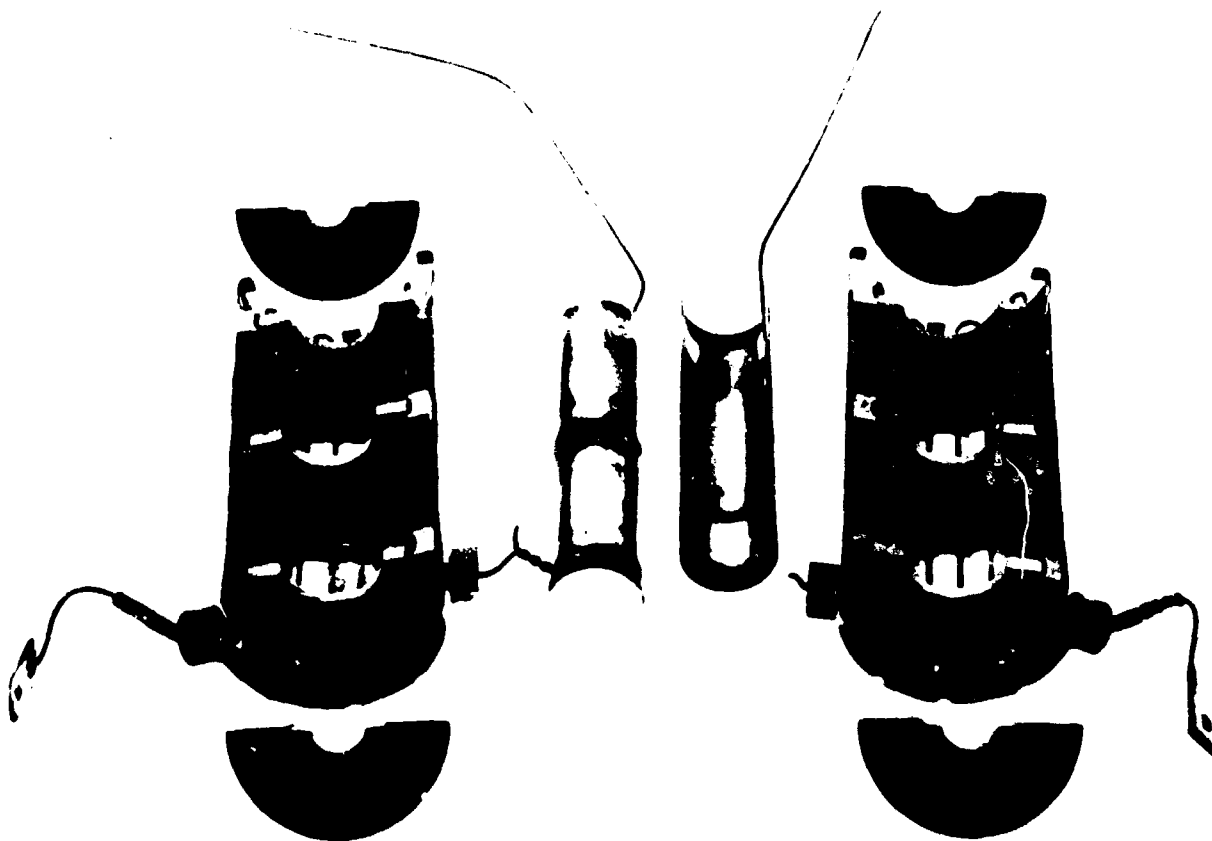


FIGURE 2. ANNEALING HEATER DISASSEMBLED AFTER TEST



FIGURE 3 ASSEMBLED ANNEALING HEATER

Concentric tantalum tubes were placed around the entire length of each heater section (i.e., preheater boiler, and superheater). These acted as radiant heat shields for thermal insulation and also as an impurity getter. Figure 4 shows the main loop heaters and the radiant heat shield in place.

The vapor test section consisted of a Cb - 1.0 w/o Zr test housing, three fixed-plate orifices (one fabricated from Mo + 0.5 w/o Ti and the other two from Cb - 1.0 w/o Zr), and three Mo + 0.5 w/o Ti test coupons. Figure 5 shows the assembly of the vapor test section with the test coupons positioned directly behind each orifice. The coupons were placed at angles of  $30^\circ$ ,  $60^\circ$ , and  $90^\circ$  to the vapor impingement. The coupons were weighed and their dimensions taken before being assembled into the test section. The diameters of the orifices were also recorded prior to their assembly into the test section.

The condenser was fabricated from 5/8 inch diameter Cb + 1.0 w/o Zr tubing (Wah Chang, Heat No. 10 3186) having a wall thickness of 0.065 inch. A 3-foot length of the condenser tube was finned with 0.015-inch thick Cb - 1.0 w/o Zr circular fins spaced 0.076 inch apart. The fins were press fitted over the outside diameter of the tubing and were not bonded to the outer wall.

The surge tank that served as an accumulator and a means of liquid-level measurement was fabricated from Cb + 1.0 w/o Zr. The level measurement probes, similar to the "J" type resistance probes, consisted of copper and Cb + 1.0 w/o Zr lead wires insulated from the Cb + 1.0 w/o Zr tube wall with high-purity, high-density alumina. Four probes were placed horizontally in the area where the level was desired, and one probe was placed vertically in the surge tank. The vertical probe would read the liquid level throughout the depth of the surge tank, while the horizontal probes would read various levels at the probe position. In addition to these level probes, a "spark plug" type probe utilizing a packing gland design was fabricated from 316 stainless steel. This probe was used only to determine the liquid level during the initial filling of the loop with potassium. It was welded to the Cb - 1.0 w/o Zr surge tank by using a coextruded bimetallic joint, Cb + 1.0 w/o Zr to Type 316 stainless steel. However, the joint was not exposed to potassium or to elevated temperatures.

An inert-gas inlet to the surge tank was also provided in order that control of the condensing process could be maintained. The inlet line was Cb - 1.0 w/o Zr and connected to 316 stainless steel tube by use of a Swagelok fitting in the cool region of the chamber.

The thermal insulation used in this loop test consisted of a solid-type insulation called "Foamsil". The Foamsil is a foamed silica and is essentially 99 percent  $\text{SiO}_2$ . In the foaming process, methane (1%) and carbon monoxide (99%) gases result and are present



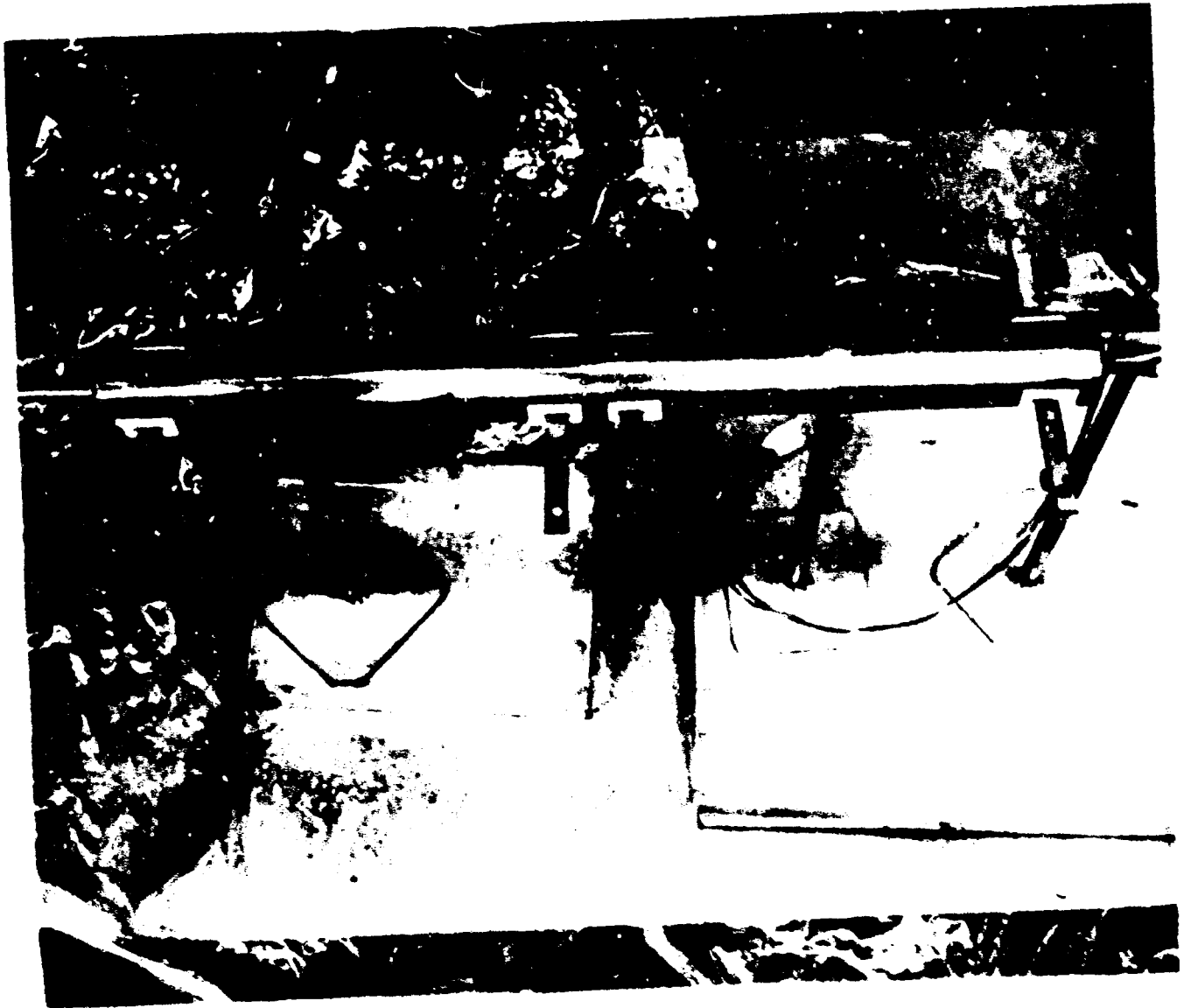
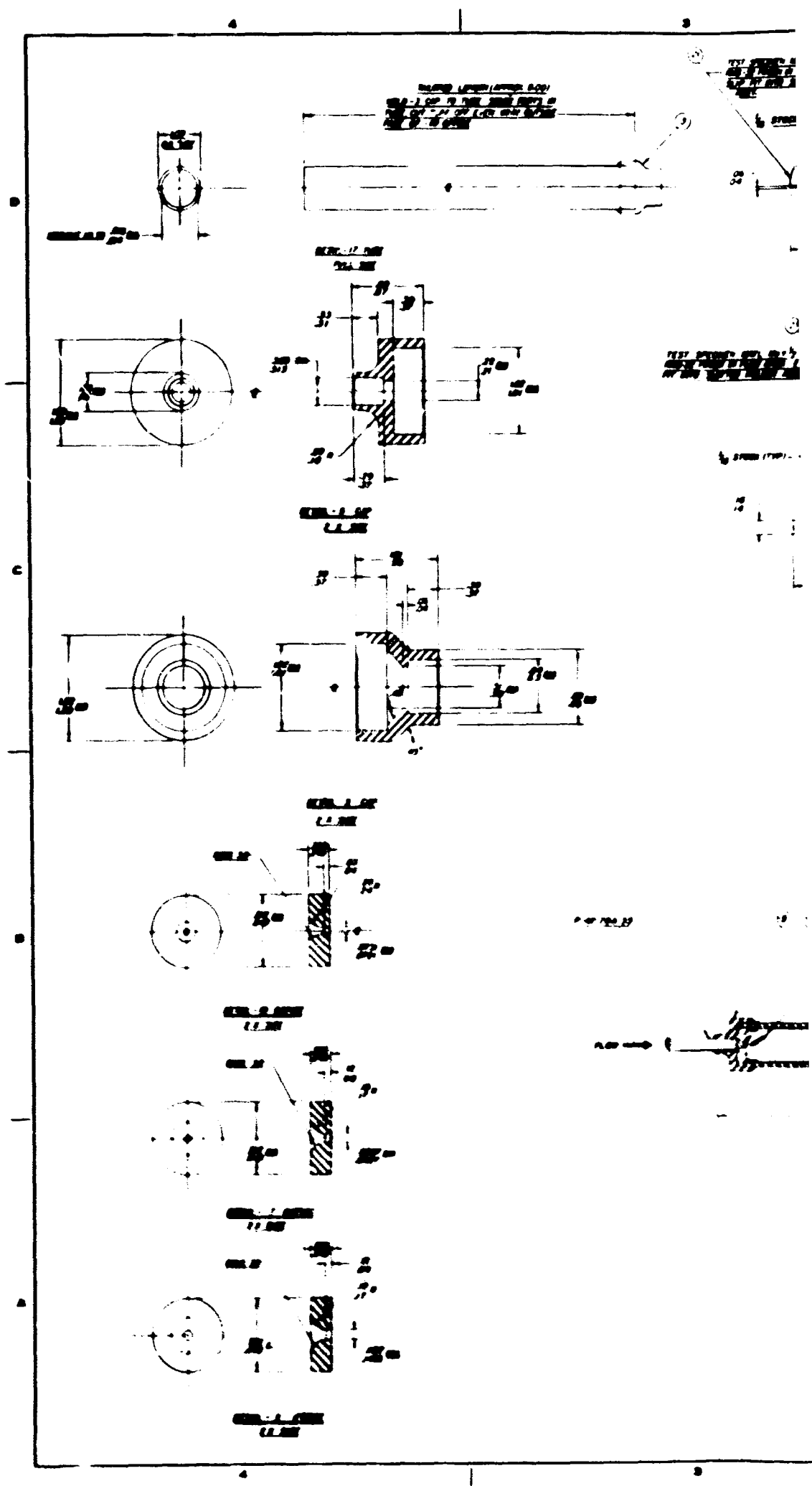
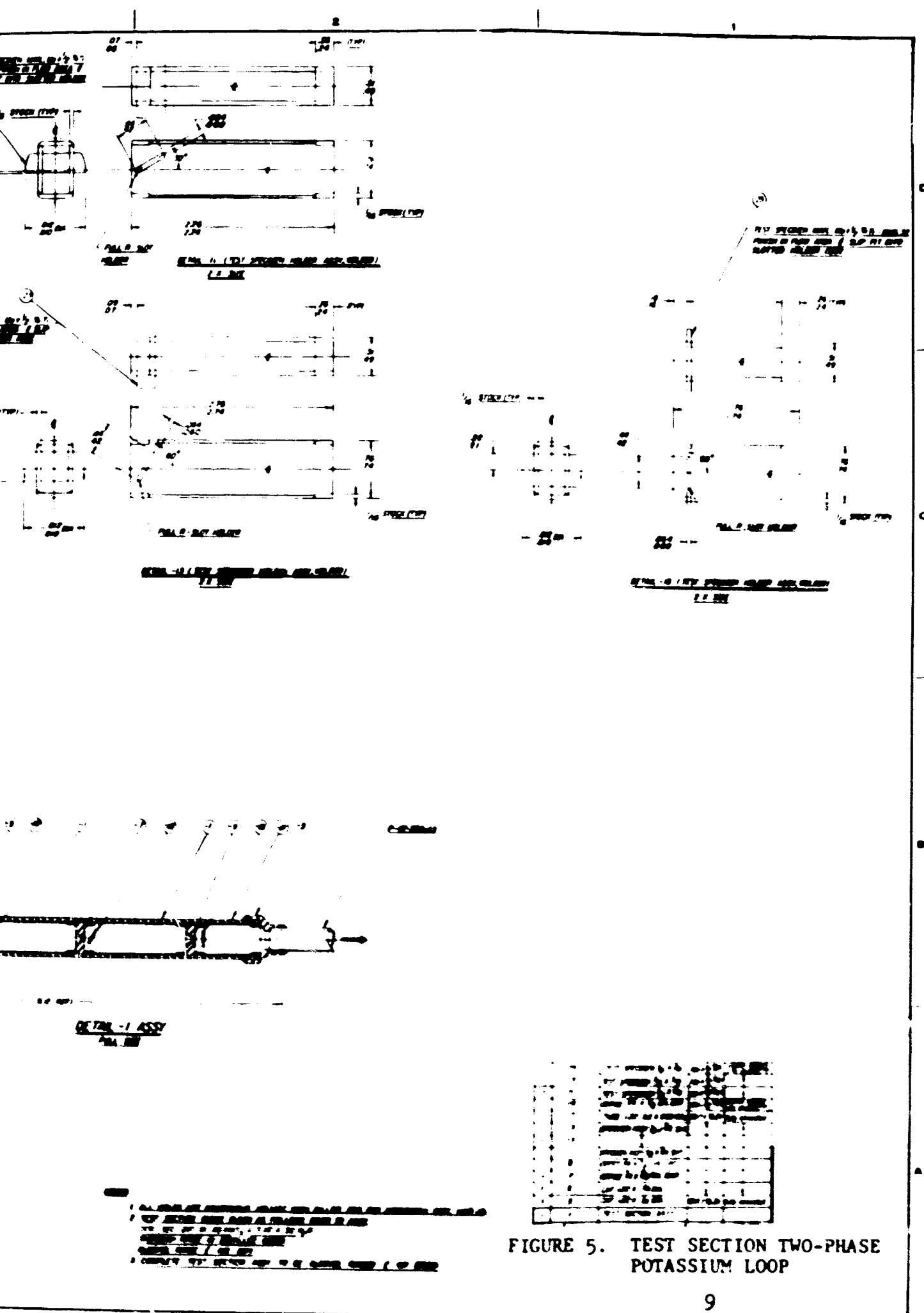


FIGURE 4. LOOP HEATER ASSEMBLY





in the isolated voids within the material. Since these are objectionable from an impurity standpoint and are a source during vacuum outgassing, the material was baked at 2000°F in a vacuum of  $10^{-3}$  torr for 12 hours and then argon-quenched. The process was repeated again, and the individual voids were disrupted. The net result was a material having a connected cellular structure rather than isolated voids. Compatibility tests with tantalum and Cb + 1.0 w/o Zr were conducted at 2000°F and a vacuum of  $10^{-5}$  torr to determine the proper heat treatment for impurity removal from the Foamsil material.

Double radiant shields were used in the heater area to reduce the heat losses. Shields were fabricated from tantalum tubing.

The electrical insulation consisted of conventional alumina insulators; however, since the heaters were specially fabricated, special electrical insulation had to be provided. Because of the ease of fabrication and the complex insulator shapes, boron nitride was selected for the electrical insulation in the heaters. Commercial-grade boron nitride was evaluated in tests with tantalum and Cb + 1.0 w/o Zr material at 2000° to 2200°F and in a vacuum of  $10^{-5}$  torr. Although the material indicated adequate electrical insulation, excessive contamination was noted on the refractory metals used in the test. As a result of these tests and additional testing, it was found that the boron nitride had to be heat-treated at a temperature of 2000°F and in a vacuum of at least  $5 \times 10^{-6}$  torr for 15 hours. An outgassing curve showed that the bakeout time of 15 hours was adequate to remove any vapor residuals within the material. The various insulators were machined to design, heat-treated in a vacuum to remove contaminants, and stored in plastic bags containing high-purity argon. Future handling of these parts required clean, lint-free cotton gloves.

The instrumentation of the loop includes thermal measurement, pressure measurement, flow measurement, and level measurement. Thermal measurement was performed on the loop through the use of Pt + 6% Rh vs Pt + 30% Rh thermocouples. The thermocouples were sheathed in 1/16-inch-diameter tantalum tubing and were insulated with high-purity alumina. Attachment to the loop was mechanical, since special thermocouple tabs were welded to the exterior wall of the loop tubing. The thermocouples were 2 feet in length and terminated to a Chromel-Chromel junction point. Readout of these temperatures as well as other temperatures was by an integrating digital voltmeter and recorder. This system could read and record 70 readouts in approximately one minute. Certain control temperatures were also read manually on a Brown recorder to facilitate the initial operation of the loop. Figure 6 shows the instrument and control panel in the remote control room.

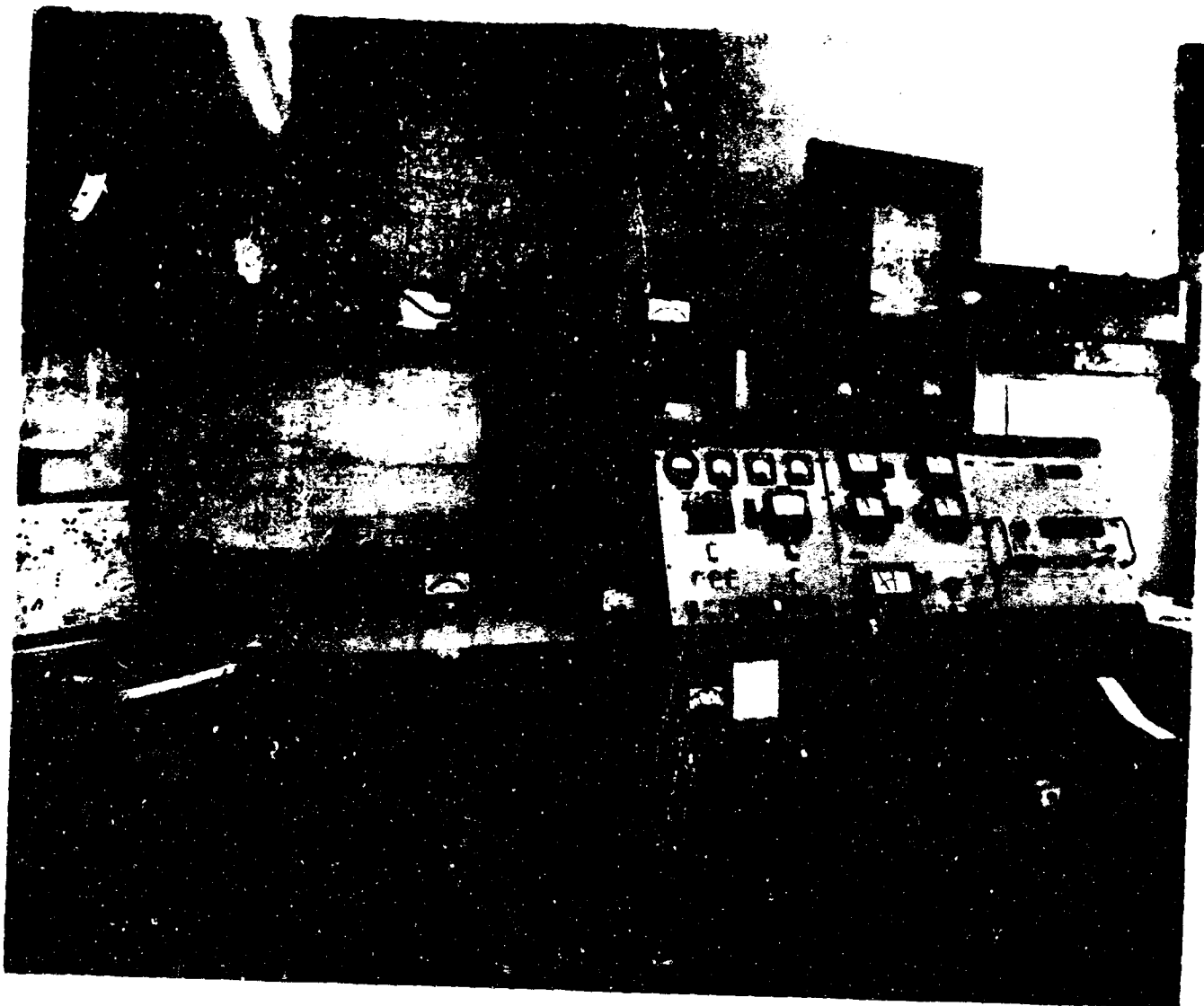


FIGURE 6. CONTROL PANEL

Heater current and voltage readouts were provided for each loop heater including the annealing heaters. In addition, power-driven Variacs controlled at the control panel were employed for the main loop heaters.

Flow measurement was made at the control panel through the use of a Hewlett-Packard vacuum-tube voltmeter. A special no-flow alarm was installed which turned off all power to the loop when triggered by the VTVM. This alarm was usually set at 50 percent of the desired flow conditions.

Loop pressure measuring the inert-gas pressure on the accumulator was read by an Ashcroft compound gauge. Potassium vapor pressure within the loop was not measured, since reliable pressure-measuring devices fabricated from Cb + 1.C w/o Zr were not available.

Readout of the modified "J" probes was through the use of a Ballentine millivoltmeter. A selector switch allowed level monitoring of any one position, and an automatic sequencing device provided the automatic readout of all positions.

Argon supply to the test was provided through the use of a central supply and associated header system. Liquid argon was used to charge the central supply. The argon was monitored for oxygen and moisture content prior to its use in the test. The argon from this system had a dew point of minus 90°F or better and an oxygen concentration of less than 8 ppm. All valves used in the metering of the argon are bellows-seal-type valves. In addition to leak-checking the valves for external leakage, each valve was leak-checked with a mass spectrograph for leakage across the valve seat.

### SECTION III - TEST CHAMBER

Since the refractory alloys, especially the Cb + 1.0 w/o Zr alloy, exhibit poor oxidation resistance at elevated temperatures and is subject to interstitial contamination, extreme care must be exercised in exposing these metals to environments for long periods of time at elevated temperature. Contamination of the Cb + 1.0 w/o Zr with the interstitials, oxygen, nitrogen, hydrogen, and carbon, usually results in a loss of desirable mechanical properties (such as ductility). A more subtle effect which may occur when contaminated Cb + 1.0 w/o Zr is exposed to elevated temperature alkali metal is a loss in corrosion resistance. Therefore, in order to prevent any contamination of the loop during testing, a special argon test chamber and an argon purification system were designed for the loop. Design of the test chamber allowed evacuation down to less than  $10^{-3}$  torr vacuum. The associated plumbing, including the argon purification system, was also designed to be evacuated to less than  $10^{-3}$  torr. The argon purification system provided a continuous purification of the test argon. A bypass system which flowed approximately 60 cfm of argon included an argon blower, molecular sieves, hot titanium chip furnaces, and associated plumbing.

The vacuum equipment used for evacuation of the chamber and associated plumbing included liquid nitrogen cold traps, a KD 30 Kenny roughing pump, a 6-inch oil diffusion pump, and a KC-5 Kenny holding pump. The pumping system was adequate to evacuate the test chamber (clean, dry, and empty, down to  $10^{-3}$  torr within a 4-hour period. Figure 7 shows part of the vacuum system relative to the test chamber.

The purification system was designed to eliminate oils, hydrocarbon, water vapor, oxygen, and nitrogen from the test argon. Two molecular sieves (one used as a spare unit) employing 1/8 inch diameter Linde Type 5A pellets, getter moisture, oils, and hydrocarbons from the argon. After the argon is passed through the sieves, it is passed through the low temperature ( $800^{\circ}\text{F}$ ) titanium filled furnace to getter the oxygen from the gas. The titanium was shredded from 0.003-inch-thick foil and placed into the furnace area. External heaters were used to heat the gas and foil to  $800^{\circ}\text{F}$ . Gas thermocouple probes monitored the temperatures in this area. Following the oxygen removal, the gas was then caused to flow through a high temperature ( $1500^{\circ}\text{F}$ ) titanium furnace to remove the nitrogen from the gas. This furnace is similar to the low-temperature furnace except for additional heater capacity. A spare system was incorporated in the purification train to permit flexibility during test operation. Figures 8 and 9 show the molecular sieves and the titanium furnaces.

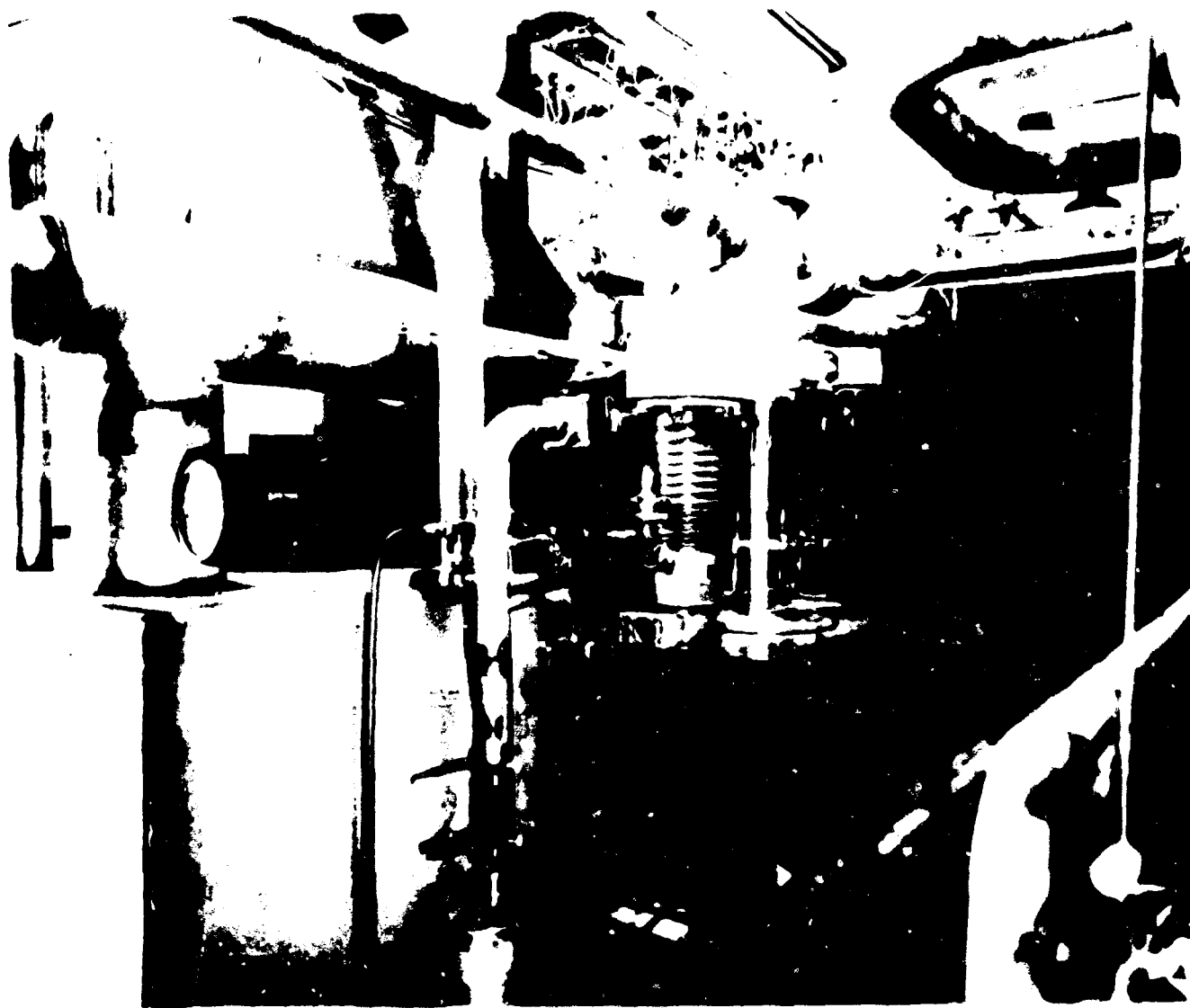


FIGURE 7. TEST CHAMBER VACUUM SYSTEM





FIGURE 8. TEST CHAMBER PURIFICATION SYSTEM



FIGURE 9. TEST CHAMBER PURIFICATION SYSTEM

Since the argon blowers were part of the test chamber system a special chamber with connecting plumbing was designed to house the argon coolant blower and the argon purification blower. Vacuum and leak-tightness specifications also existed for this portion of the system. Figure 10 shows the two blowers--the 1,200-cfm Buffalo argon coolant blower (on top) and the 60-cfm Hoffman argon purification blower. The Hoffman No. 4010 blower is a 10-stage centrifugal blower which was directly coupled through a heavy-duty flexible coupling to a 5 horsepower, 3,600-rpm, 440-volt, 60-cycle, 3 phase, high temperature motor. Both units operated with grease-packed bearings lubricated with Dow Corning silicone grease No. 44. Provisions were made to lubricate the Buffalo blower externally during its operation. In addition, a Varidrive set was attached to the Buffalo coolant blower so that the coolant flow could be adjusted. The drive mechanism was a Reliance V-S Drive, V-S Straton Style S, 5 horsepower, 460-volt 60-cycle, 3-phase Type 25F37 with a speed range from 2,500 to 3,700 rpm.

Since the operation of the blower bearings was limited to 200°F, provisions had to be made to cool the test chamber outlet gas. A water-to-argon heat exchanger was placed in the outlet gas line to extract the heat from the argon and permit the lower bearing temperatures. The heat exchanger was a tube-shell design.

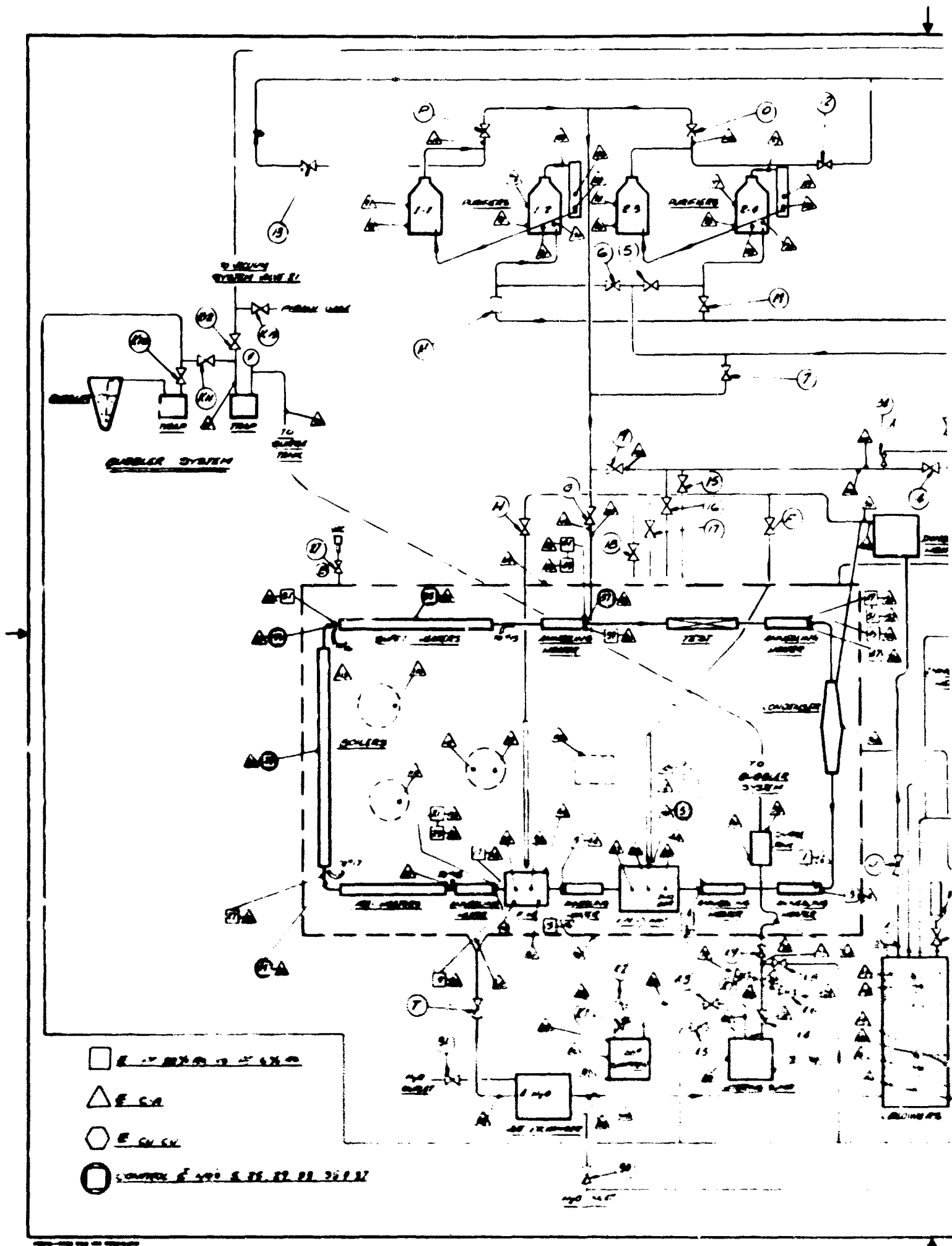
Figure 11 shows the schematic of the test chamber and the associated plumbing. All valves were bellows seal valves, and those incorporated in the purification system had a 1600°C capability.

The argon system was monitored through the use of a CEC Type 26-303 moisture monitor, a Beckman Model 80 Trace Oxygen Analyzer, and a modified CEC Type 21 611 Residual Gas Analyzer (RGA). The residual gas analyzer could give impurity content for the impurity mass numbers from 0 to 80. The oxygen concentration in the test argon could be monitored continuously during test, however after the test was started, periodic samples were taken to conserve on argon. Because the RGA was designed to operate in vacuums it was modified to screen pressure systems. The modification was satisfactory for qualitative results but could not be extended to yield quantitative results. The impurity level that was considered adequate for the 1,000-hour test was 1 ppm O<sub>2</sub> and a moisture content of less than 5 ppm.

The instrumentation of the test chamber involved temperature measurement of the inner chamber walls during the vacuum bakeout procedure and during test, and provisions for feedthrough for the electrical and thermocouple leads. Chromel-Alumel thermocouples were used to measure the skin temperatures within the chamber. Standard Conax vacuum feedthroughs were used for both the power leads and the thermocouple wire. Figure 12 shows part of the two instrument ports containing the feedthroughs and the various power feedthroughs for the heater system.



FIGURE 10. PURIFICATION BLOWER SYSTEM





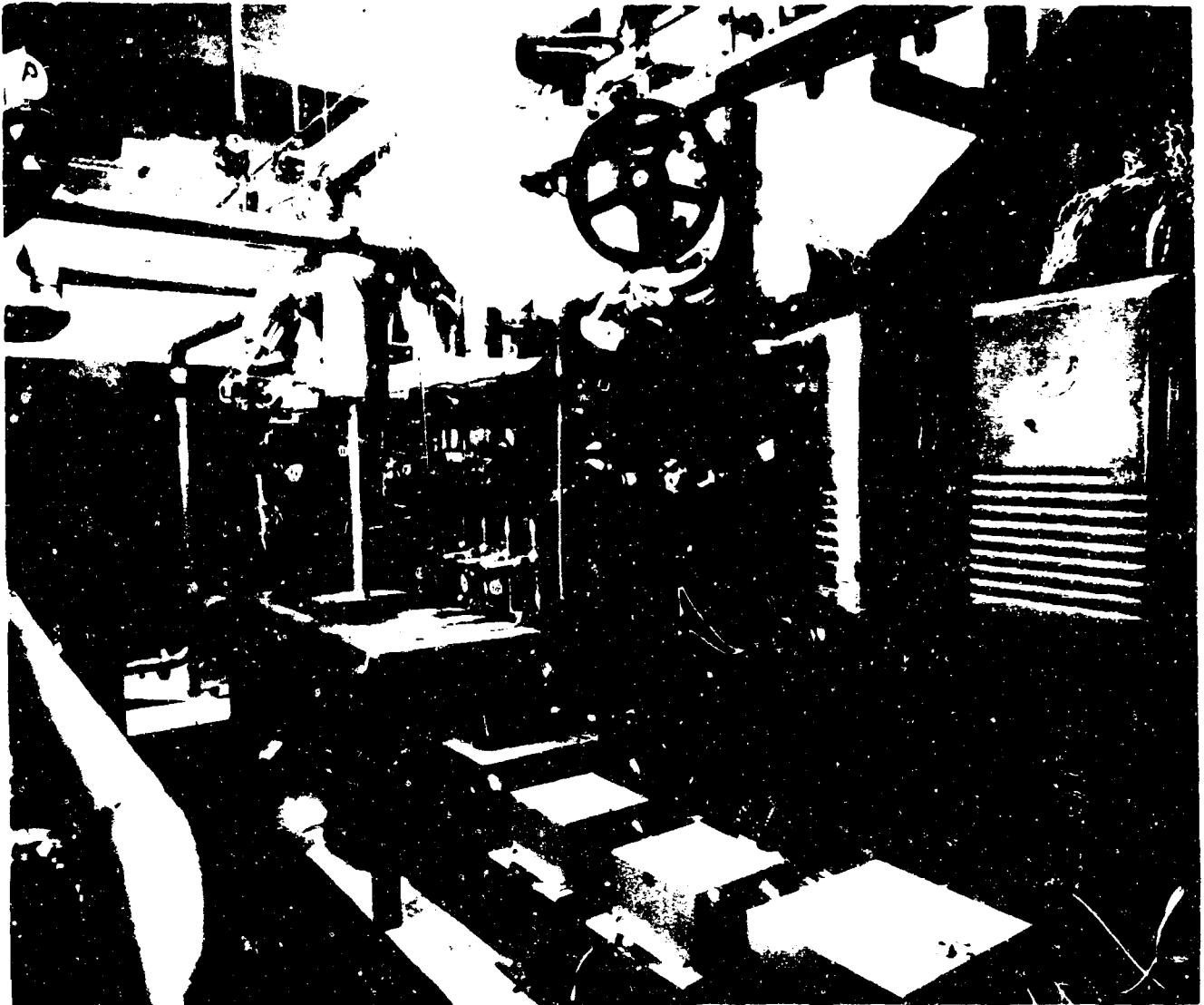


FIGURE 12. BACK VIEW OF TEST CHAMBER SHOWING INSTRUMENTATION PORTS

## SECTION IV - MATERIALS

Where possible, the materials employed in this test were tabulated as to heat number, past metallurgical history, and chemical analysis before being applied to the test apparatus. The Cb + 1.0 w/o Zr alloy was purchased to purity specification. Although higher purity was desired, material having lower impurity content was not available. This is especially true for the loop tubing. The following maximum impurity concentration in the Cb + 1.0 w/o Zr material allowed was: 300 ppm oxygen, 300 ppm nitrogen, 100 ppm carbon, and 50 ppm hydrogen. The columbium alloy was obtained in a stress-relieved condition (1 hour at 2000°F). The Mo + 0.5 w/o Ti was also purchased in the stress-relieved condition (1 hour at 1800°F).

Ultrasonic inspection of the columbium alloy tubing was made prior to loop fabrication. All tubing ordered for the loop passed the inspection.

The stainless steel required for the test chamber and plumbing was purchased according to ASTM and AMS specifications.

All metal raw material such as the Mo + 0.5 w/o Ti, Cb + 1.0 w/o Zr, Type 316 stainless steel, and other stainless steels were cleaned according to AiResearch Cleaning Specification C-57, titled, "General Procedures for Cleaning Components and Systems That Carry Liquid-Alkali Metals."

The thermal insulation, Foamsil, was purchased as a standard commercial item and was specially processed prior to its usage in the loop test.

The boron nitride raw material was also purchased as a standard item; however, high-purity boron nitride was specified. Composition of the material was 97% boron nitride, 2.4% boron oxide, 0.2% alumina and silica, and 0.1% alkaline earth oxides. This material was also specially processed prior to being used as an electrical insulator.

The potassium that was used in this test was the special high-purity material. The potassium (Sample PDA-2) analyzed 8 ppm oxygen and had the following trace metal analysis (ppm): 70 Fe, <10 B, <5 Co, 5 Mn, 5 Al, 3 Mg, <5 Sn, 25 Cu, <5 Pb, 25 Cr, 25 Si, <5 Ti, 10 Ni, <3 Mo, <1 V, <1 Be, <1 Ag, <10 Zr, and 60 Na.



## SECTION V - LOOP FABRICATION

The loop fabrication primarily involved welding of the components and their subsequent assembly into a loop. Because of the sensitivity of the Cb + 1.0 w/o Zr to contamination when heated to elevated temperatures, care must be exercised when joining this material. The method of joining employed in the fabrication of the loop was the tungsten inert-gas (TIG) method. The two methods of providing the required environment to minimize contamination were (1) welding in a vacuum gloved dry box and (2) welding in a plastic bag that had been purged with inert gas. The vacuum dry box was used principally on component fabrication and those items that would physically fit into the dry box. The plastic bag was employed in loop assembly where field-type welds were required.

The vacuum dry box prior to welding was evacuated a minimum of three times and backfilled with special high-purity argon. After the last argon backfill, a zirconium wire was direct-resistance-heated to approximately 1200°F to getter the argon welding environment for a 1-hour period. Test coupons of Cb + 1.0 w/o Zr were then welded, and bend tests were conducted on the welded coupons. If the bend tests showed no visual cracking, the welding of the test hardware began. If the coupons indicated embrittlement, the process of evacuation and backfilling was repeated. Upon completion of the welding operation (usually 1 to 2 hours in duration), another set of test coupons was welded. The test coupons were identified as to the particular loop welds and stored for future use. A typical analysis of the parent material to be welded and a weldment is as follows:

	<u>ppm/weight</u>			
	<u>O<sub>2</sub></u>	<u>H<sub>2</sub></u>	<u>N<sub>2</sub></u>	<u>C</u>
Material prior to welding	281	<1	97	80
Welded material	281	<1	107	83

As seen from the above analysis, the method of quality control employed during the component fabrication was adequate to insure proper weldments in the Cb + 1.0 w/o Zr material.

When fabrication of the loop components was completed, a dust-free room was installed to provide a dust-free loop assembly area. This room was a wood frame with heavy-gauge plastic material for walls and ceiling. A filter and an air-circulation blower was provided in the room, and the room was slightly pressurized to keep the possible exterior contaminants out of the assembly room. A working bench was installed and a plastic material (0.0025 inch thick and heat-sealable on one side) for the

welding bag was placed on the work bench. The necessary equipment such as a weld filler rod and welding tools were placed on the work bench. Figure 13 shows the components in place and the plastic bag ready for fabrication. As soon as the bag was fabricated, inert gas was passed through the bag by means of evacuating with a vacuum pump and simultaneously purging with inert gas. A mixture of argon and helium was initially passed through the bag. This permitted a leak check of the plastic envelope with a helium mass spectrometer. Although the helium can diffuse through the plastic material, the mass spectrometer could be set at a less sensitive scale and still be useful in detecting leaks in the plastic bag. As soon as the leak check was completed, the helium and argon purging was continued for approximately 2 hours and a final purge of high-purity argon was made. As in the vacuum dry-box welding, test coupons of Cb + 1 w/o Zr were welded and bend tests performed on the welded coupons prior to and after welding the loop. A typical analysis of a Cb + 1.0 w/o Zr weldment is as follows:

	<u>ppm/weight</u>			
	<u>O<sub>2</sub></u>	<u>N<sub>2</sub></u>	<u>C</u>	<u>H<sub>2</sub></u>
Material prior to welding	230	90	50	2
Material welded	260	109	50	1

As seen from the above analysis, a very slight pickup of oxygen and nitrogen is evidenced. However, the total content is below the impurity level established for procurement of the raw material. Since it is recognized that this method of fabrication does permit some contamination, all loop welds (a total of eight) performed with this technique were scheduled for annealing at 2200°F for one hour. Figure 14 shows the loop being welded within the plastic bag.

As soon as the loop was assembled, it was attached to a metal frame and placed in an upright position. As shown in Figure 15, the weldments were radiographed, and the loop was leak checked with the helium mass spectrometer. After passing these tests, the loop was triple-wrapped with 0.003-inch-thick tantalum foil, which acted as a contaminant getter. Upon completion of the tantalum wrapping, the heaters and the double radiant heat shields were installed on the loop. Since fusion tack welds were required, individual welding plastic envelopes were employed. Similar techniques used in the loop fabrication were employed in the heater installation. Figure 4 shows the completed preheater section consisting of two heaters and a set of radiant shields. Figure 16 shows the assembled heater section of the loop. This

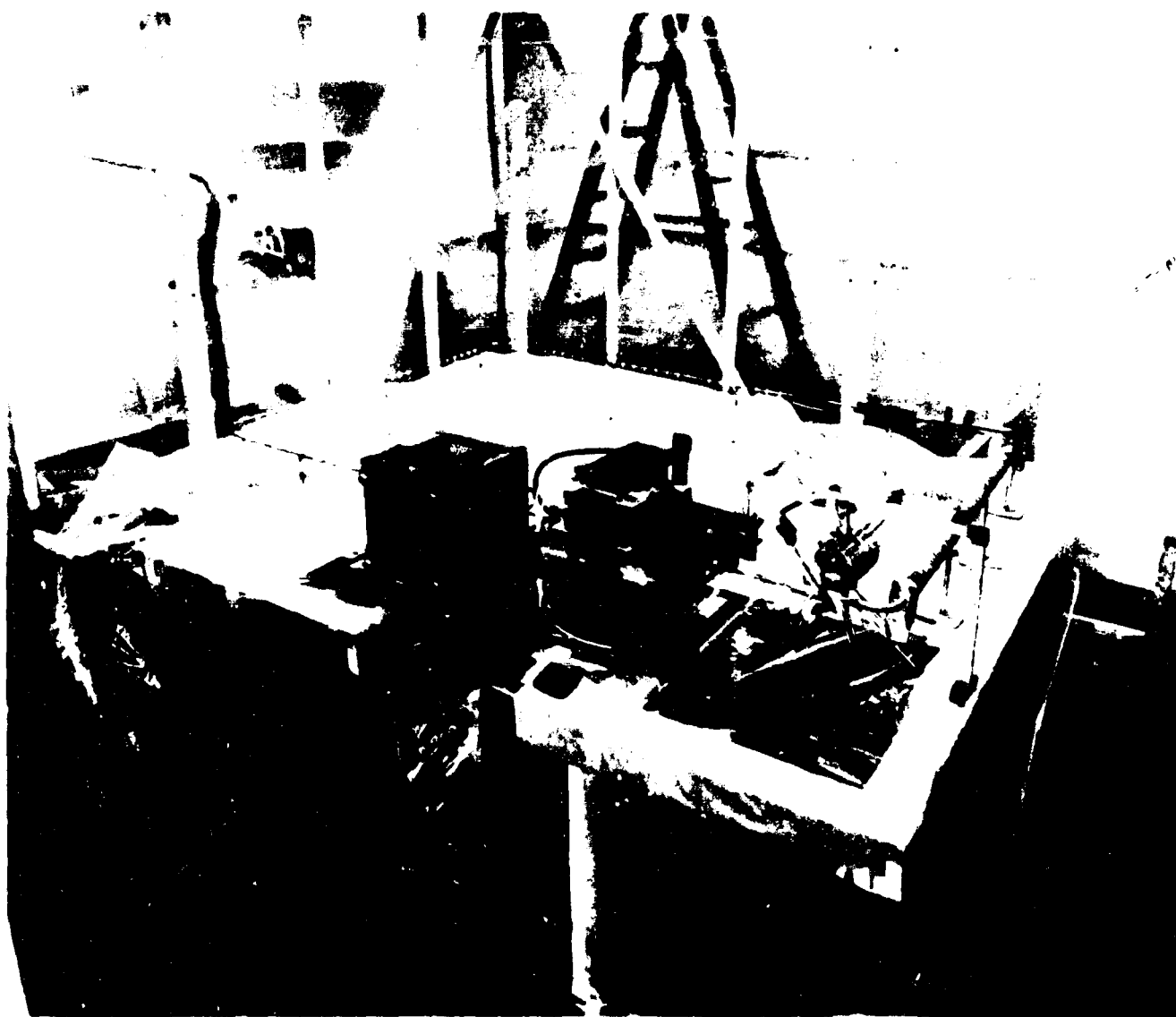


FIGURE 13. ASSEMBLY OF LOOP COMPONENTS FOR LOOP FABRICATION



FIGURE 14. LOOP FABRICATION WITHIN PLASTIC ENCLOSURE

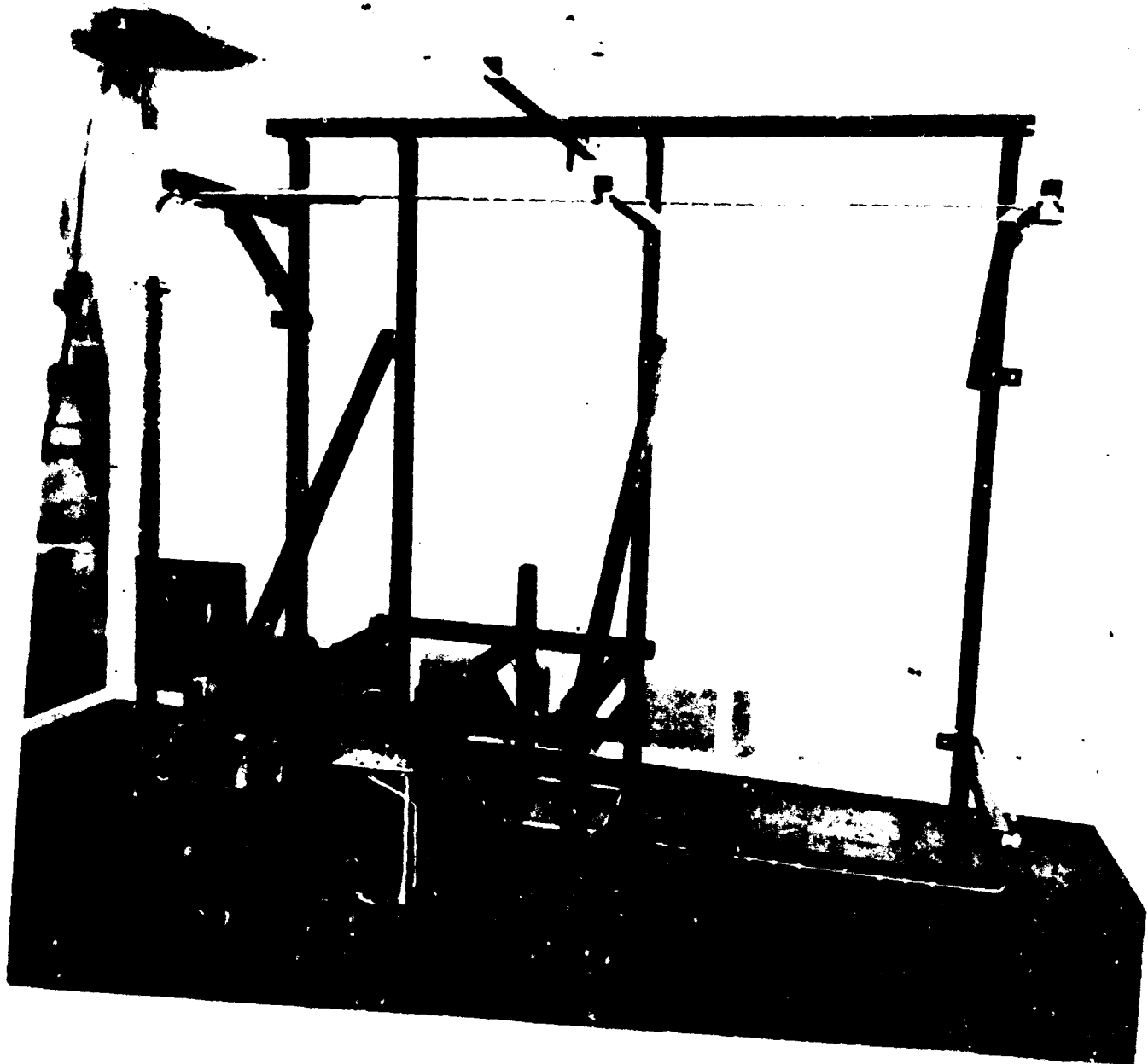


FIGURE 15. FABRICATED LOOP PRIOR TO TANTALUM FOIL WRAPPING

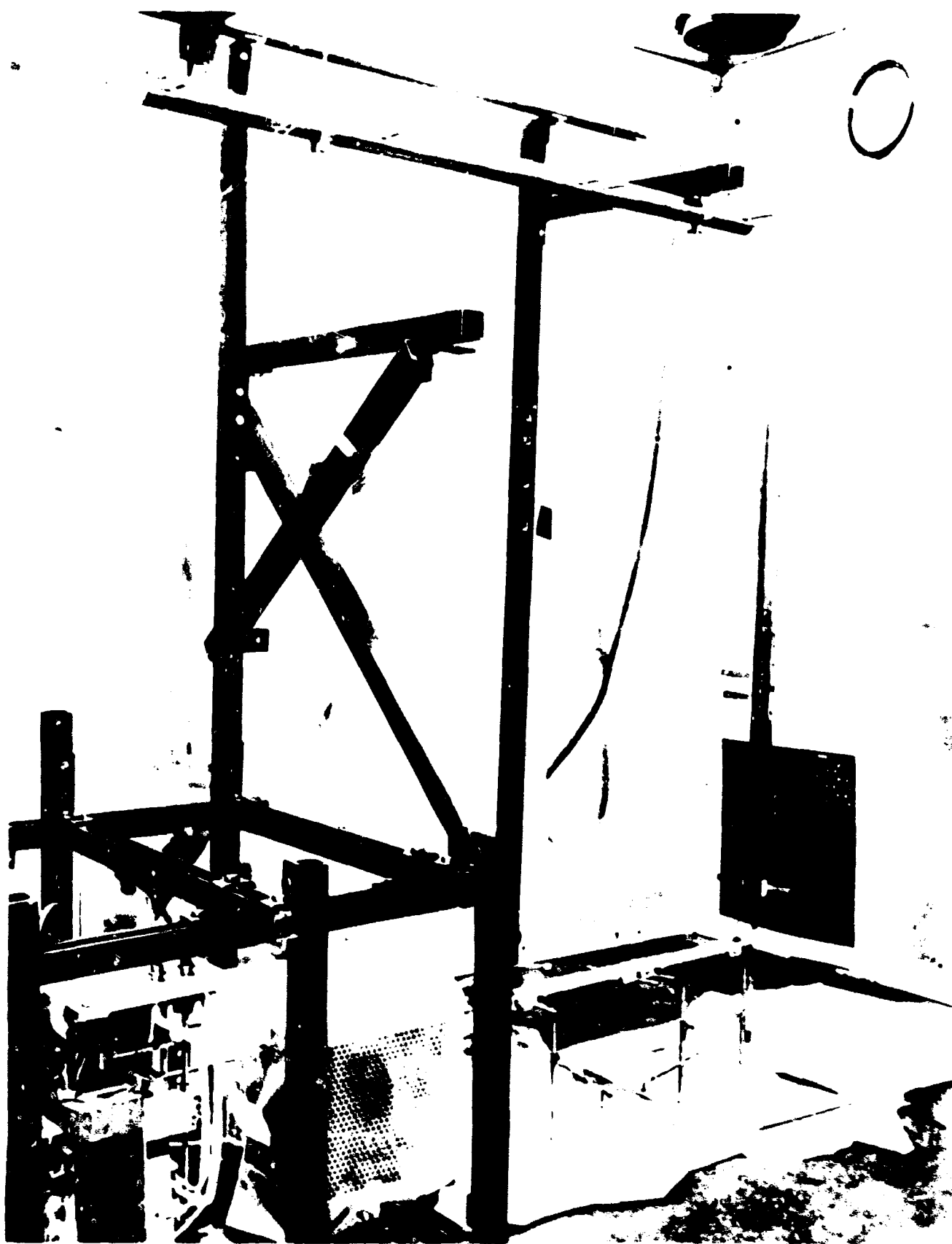


FIGURE 16. MAIN LOOP HEATERS INSTALLED ON LOOP

completed the loop fabrication; subsequently, the loop was installed in the test chamber as shown in Figure 17. To prevent dust contamination, a plastic room similar to the assembly room was fabricated at the opening of the test chamber. Electrical and instrumentation assembly and hookup was performed in the dust-free environment. The handling of the loop during fabrication and assembly necessitated the use of clean, lint-free gloves to reduce the probability of contamination.

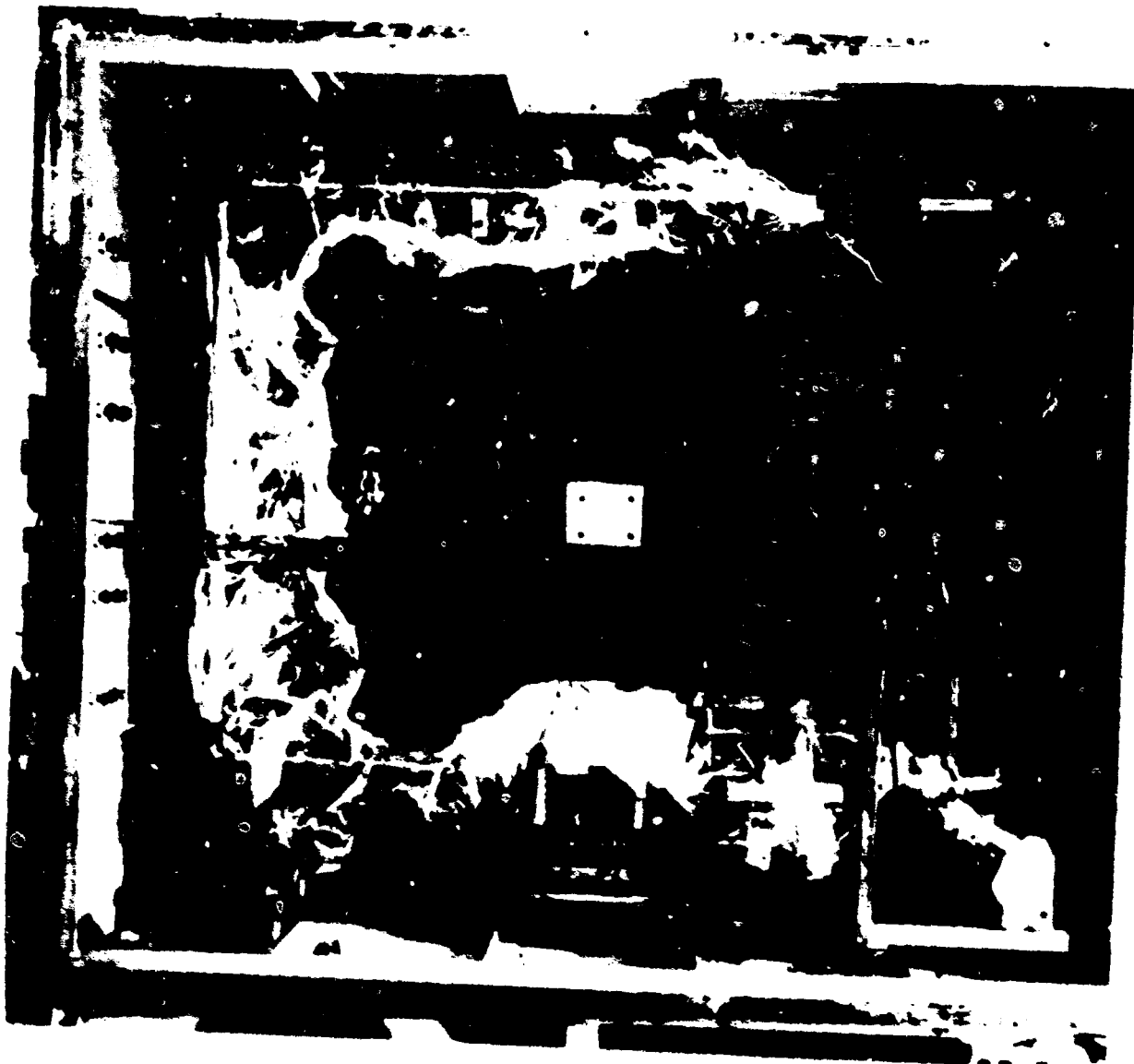


FIGURE 17. TWO-PHASE LOOP INSTALLED IN TEST CHAMBER



## SECTION VI - TEST PREPARATION

Since both the annealing and the main loop heaters were of special design and construction, their performance under test conditions had to be evaluated prior to test. In addition to the heaters, the method of liquid-level detection during loop operation had to be evaluated.

Two heater tests were conducted to determine if any problems were evident in the heater design. The first test involved a half heater section which was heated in an argon test chamber. The test chamber was evacuated to less than  $10^{-6}$  torr and backfilled with high-purity argon three times. Figure 18 shows the heater half section in the test fixture which is attached to the top flange of the heater test chamber. The heater was direct-resistance ( $I^2R$ ) heated to  $2300^{\circ}\text{F}$  for 30 minutes. No hot spots were noted in the bus bar attachment, and current and voltage measurements showed no degradation. Heating appeared to be uniform throughout the length of the heater. Consequently, the heater temperature was raised to  $3060^{\circ}\text{F}$  and held at this temperature for a period of 1 hour. Heater temperatures were taken with an optical pyrometer. Again the heater half performed satisfactorily.

Upon completion of this test a heater mock-up test was conducted. This test included a full heater assembly and an annealing (or adiabatic wall) heater. To simulate actual loop heating conditions as close as possible, the heaters were placed around a Haynes Alloy 25 tube, and air was passed through the tubing during the test. Figures 19 and 20 show the heater assembly (attached to the top flange of the heater test chamber) prior to the test. The annealing heater is shown on the left and the main loop heater on the right. The heater assembly in both heaters duplicated exactly the design of the heaters which were to be used in the loop test. The holes in the tantalum outer shell of both heaters permitted the use of an optical pyrometer to measure the temperature of the heater element. After the heaters were installed in the test chamber, the test chamber was leak-checked with a helium mass spectrometer. The chamber was then evacuated down to  $10^{-6}$  torr and backfilled with high-purity argon three times. An argon pressure of 4.5 psig, which is the operating pressure within the loop test chamber during loop operation, was maintained. The annealing heater was brought to  $2680^{\circ}\text{F}$  and held at that temperature for 2 hours. The heater performed satisfactorily under these test conditions.

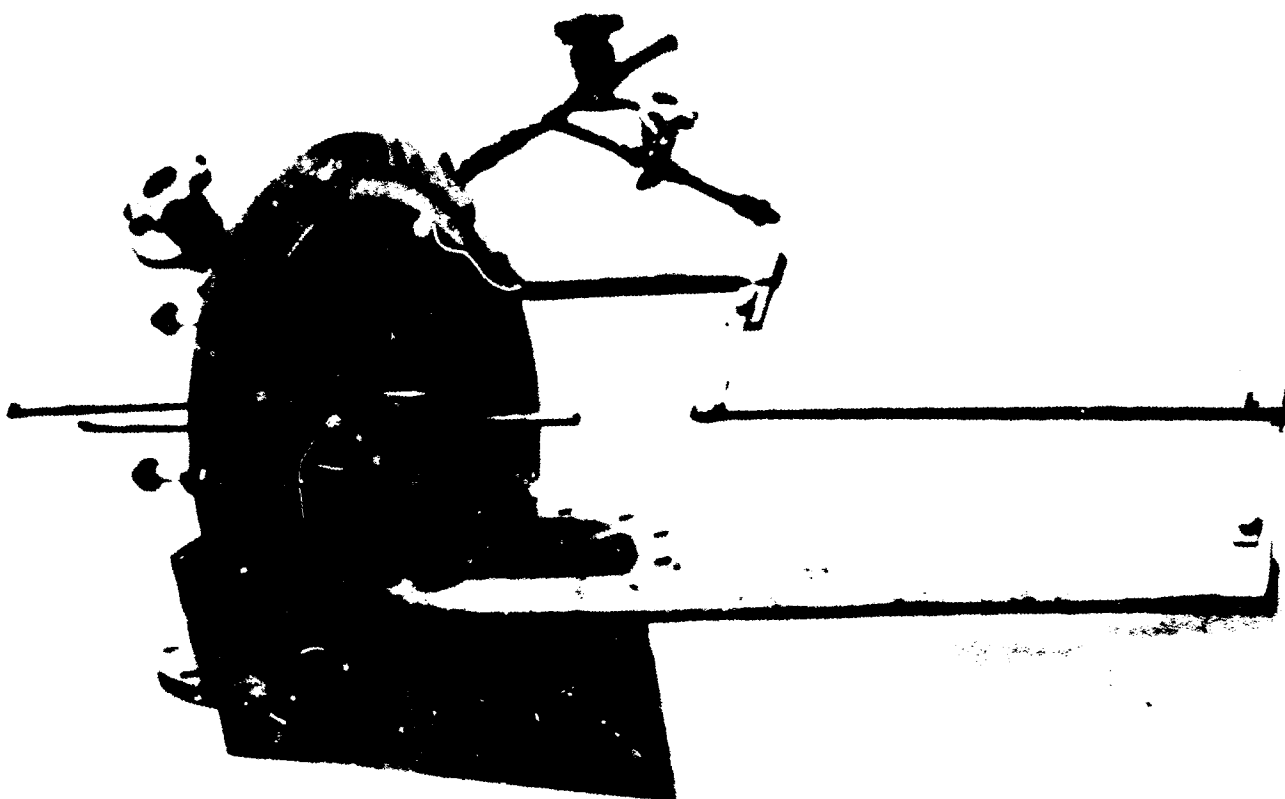


FIGURE 18. TANTALUM HEATER ASSEMBLED IN TEST FIXTURE



FIGURE 19. TANTALUM HEATER ASSEMBLY

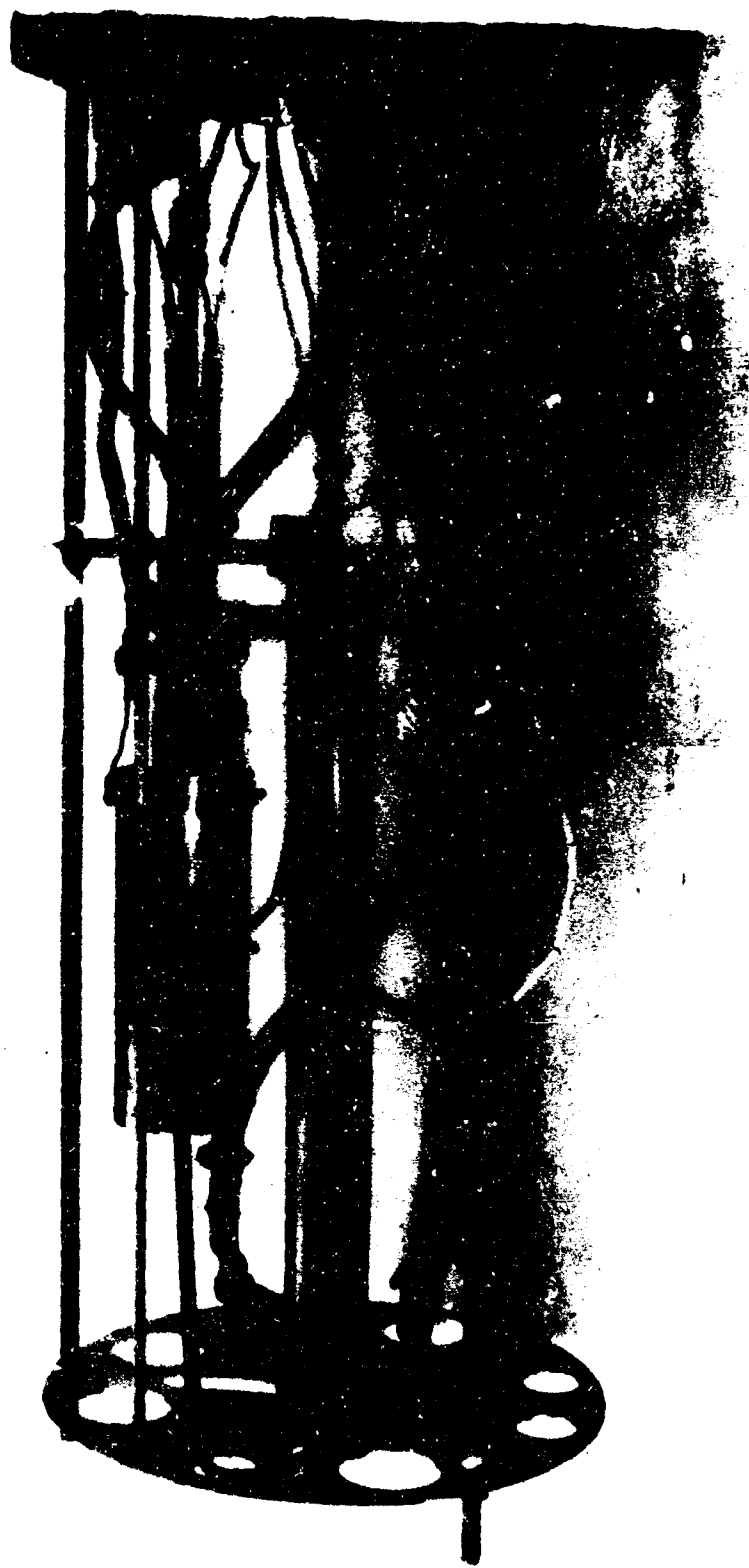


FIGURE 20. TANTALUM HEATER ASSEMBLY

The main loop heater was operated for 40 hours with the heater element temperature at 2300°F and tube temperature in excess of 2000°F. The heater performed satisfactorily, and the test was terminated. Upon removal from the test chamber, the visual appearance of the tantalum heater and tantalum radiant shields showed discoloration, which is indicative of contamination. On the tantalum areas directly underneath the boron nitride, a glazed film was evident. The tantalum heater element wire and the tantalum heater tube element were brittle--also indicative of contamination. An analysis of the tantalum before and after test showed the following results:

<u>Element</u>	<u>As Received</u>	<u>After Test</u>
O <sub>2</sub>	30	1,000
N <sub>2</sub>	22	1,250
C	38	92
Fe	8	130
B	--	700

Although the test was successful from an electrical design standpoint, it was evident that a contamination problem existed. Further testing in a vacuum bell jar revealed that the contamination resulted from the boron nitride. Additional testing on boron nitride which was outgassed at 2000°F for 15 hours showed that the outgassed boron nitride could be used satisfactorily. Figures 21 and 22 show the disassembled heaters after test and the resultant contamination.

Since the proposed design of the liquid-level-measuring device was a modified version of an existing nonrefractory-metal liquid-level probe (the "J" tube resistance-type probe), a test was required to evaluate the ability of probes fabricated from Cb + 1.0 w/o Zr to detect potassium levels. A chamber fabricated from Cb + 1.0 w/o Zr containing the probes was constructed as shown in Figure 23. An enclosed stainless-steel bottom sump containing the potassium was attached with Swagelok connections to the Cb + 1.0 w/o Zr chamber. By means of pressurizing and evacuating the volume above the potassium in the sump, the level of potassium in the instrumented Cb + 1.0 w/o Zr chamber could be varied. The liquid-level-probe test, as shown in Figure 24, was conducted in the vacuum gloved dry box which was evacuated and backfilled with argon three times. The temperature at which the tests were conducted was in the range of 200° to 263°F. The potassium level could be observed visually in addition to the probe detection signal output. All probes, including the vertical probe, were set up as the fourth leg of a Wheatstone bridge, and

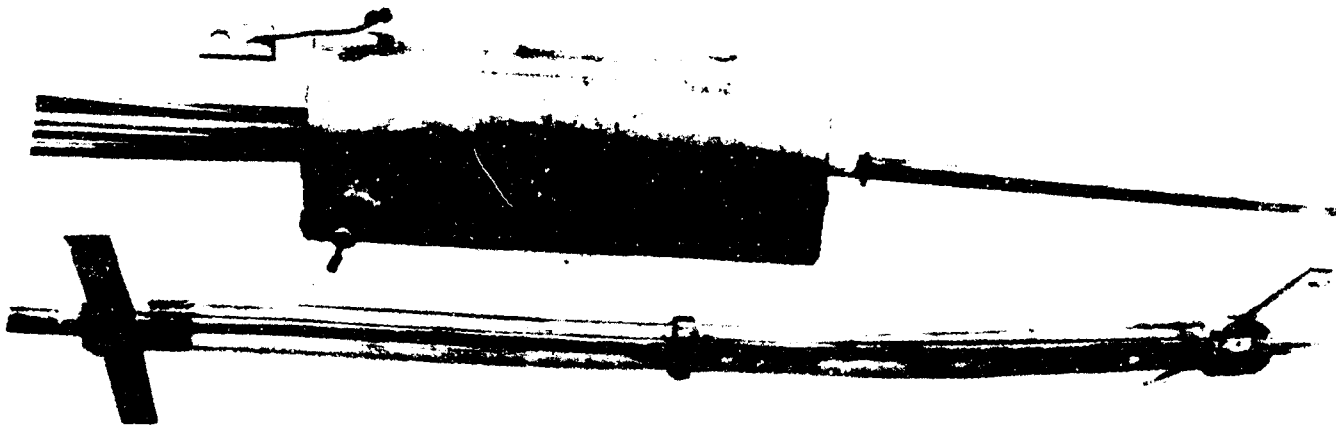


FIGURE 21. TANTALUM LOOP HEATERS AFTER HEATER TEST

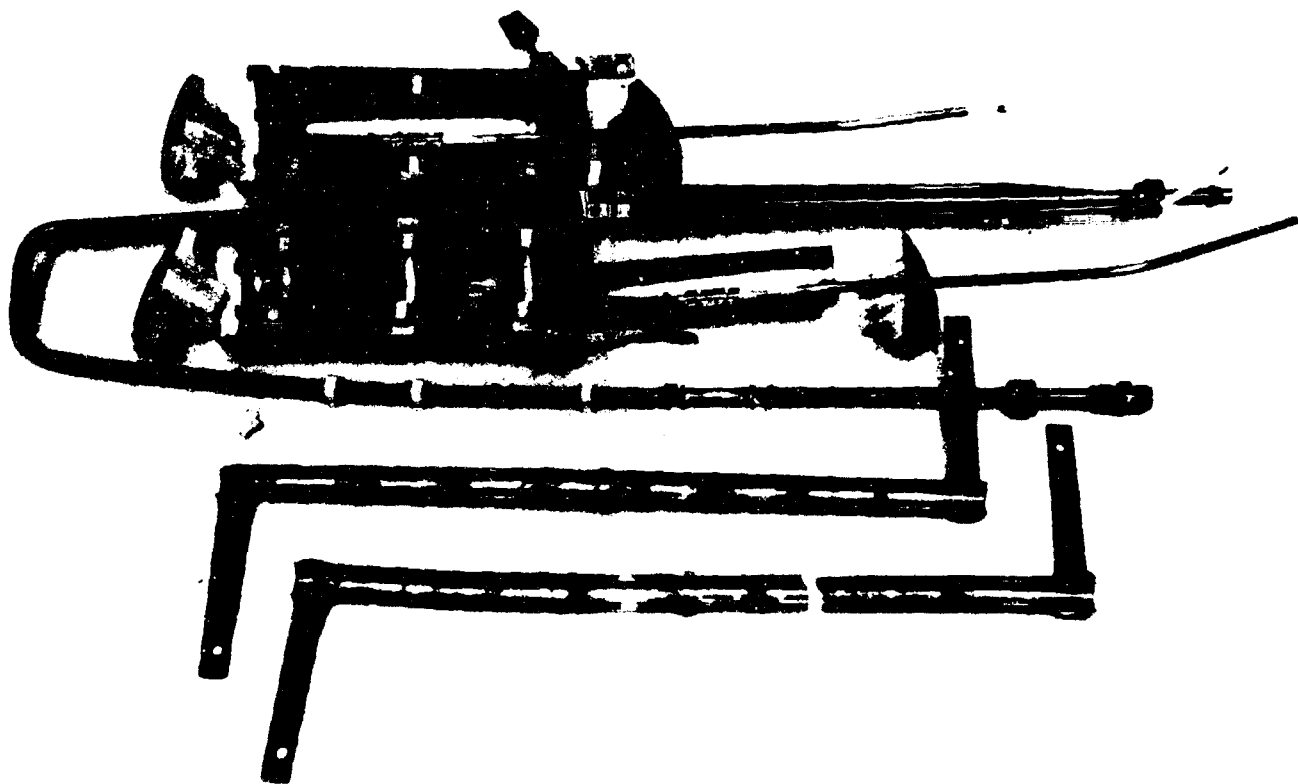


FIGURE 22. DISASSEMBLED TANTALUM LOOP HEATERS AFTER HEATER TEST

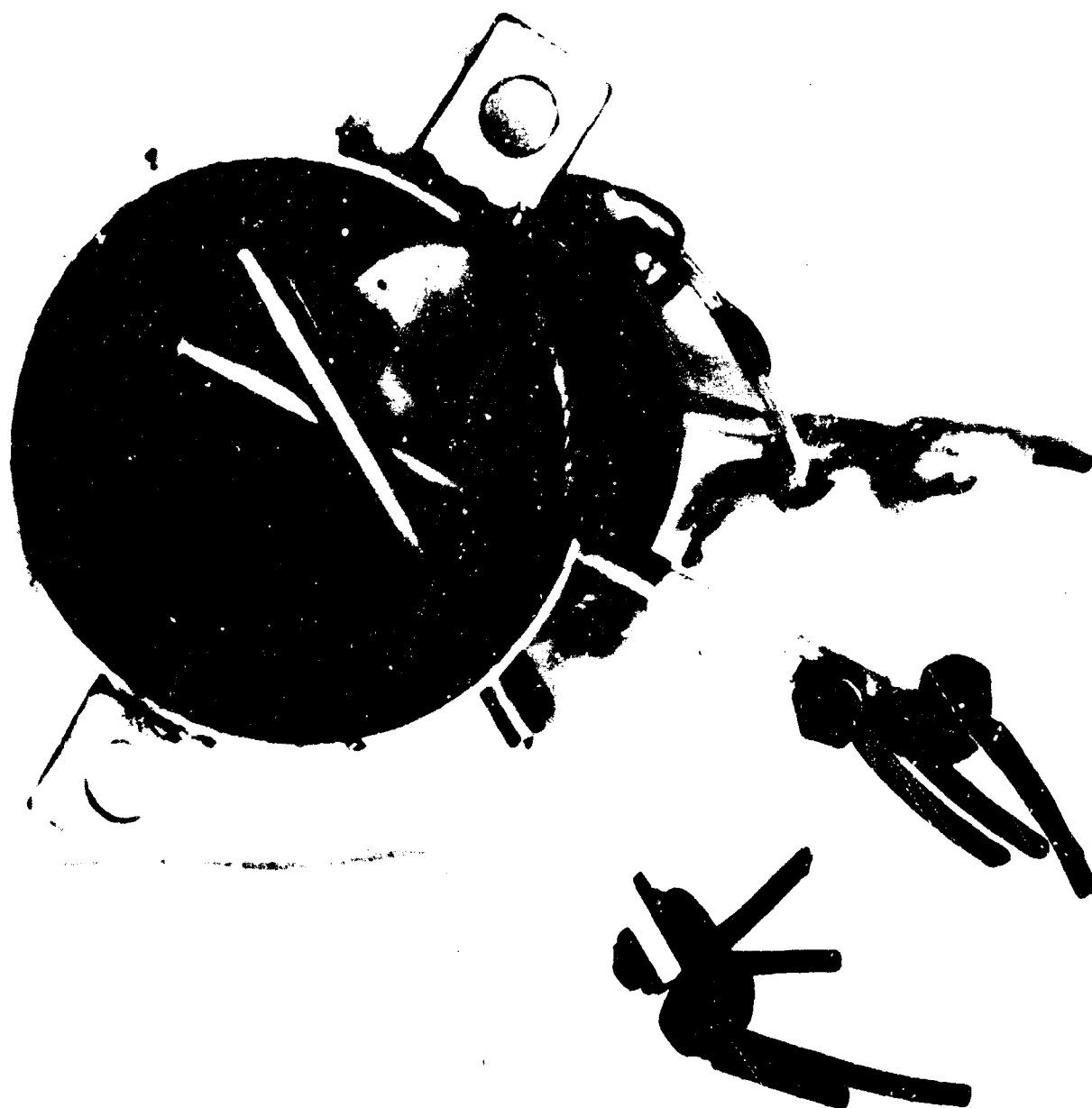


FIGURE 23. LIQUID-LEVEL TEST APPARATUS



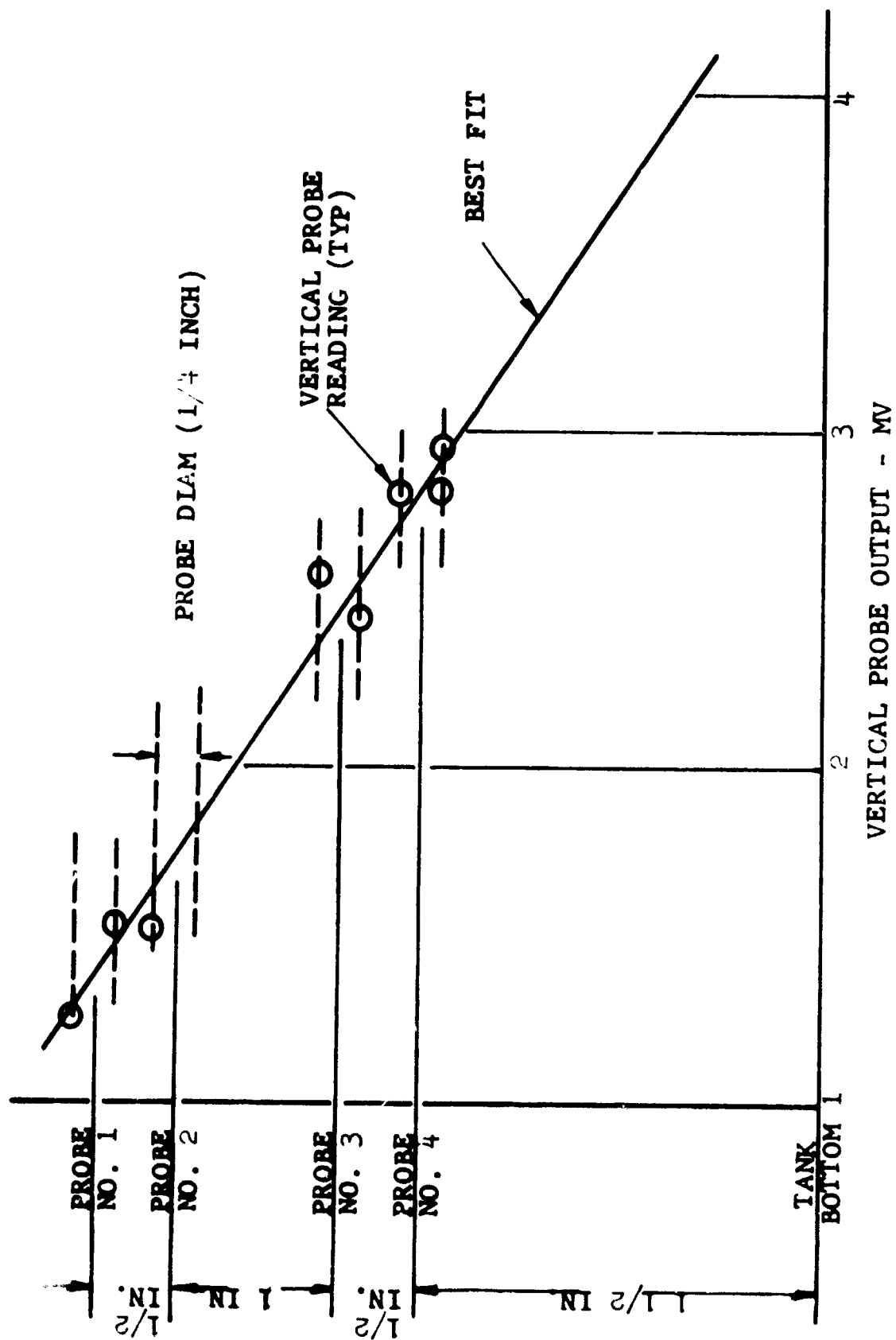


FIGURE 24. LIQUID-LEVEL-PROBE TEST SETUP

the unbalance voltage was read out on a Ballentine millivoltmeter. Two amperes of exciting current were supplied to each probe. The test consisted of slowly raising the liquid level until it touched a horizontal probe, stopping to read the millivolt output from the vertical probe, and repeating the process for the other three probes. It is of interest that probe contact with the potassium was established from the signal outputs before it could be determined visually. Figure 25 shows the results of five successive calibrations.

Upon completion of the loop installation in the chamber and the subsequent checkout of the loop electrical hookup and instrumentation, the chamber door was put in place on the chamber. Although the components of the environmental system had undergone leak-checking with a helium mass spectrometer and vacuum decay curves were obtained, the entire system with the loop installed was again leak-checked. Upon passing the leak check test, the environmental system (test chamber, ducting, purification system, and blower chamber) and the loop were evacuated to approximately  $10^{-3}$  torr. Evacuation time was minimized, since most of the components were evacuated several times prior to the system evacuation. As soon as the vacuum was achieved in the test chamber, external heaters placed on the chamber walls and ducting were turned on. Quartz lamps, infrared bulbs, and Chromalox strip heaters were employed. Figures 26 and 27 show the external bakeout heaters in place. After an initial heating period with external heating only, the loop heaters were turned on and were never allowed to exceed  $350^{\circ}\text{F}$ . Skin temperatures, external and internal, were taken during the bakeout of the system. Temperatures varied from  $250^{\circ}$  to  $300^{\circ}\text{F}$  during the bakeout. Bakeout of the box required approximately 1 week. Vacuum decay curves of the chamber while at bakeout temperatures indicated that outgassing was negligible. These curves were compared to the decay curves obtained with the system clean, dry, and empty.

As soon as the bakeout was achieved, the system was back-filled with argon gas having an oxygen content of 8 ppm. The argon blowers were operated, and the argon circulated through the system. The argon was monitored for impurities with the moisture monitor, the oxygen analyzer, and the residual gas analyzer. Figure 28 shows the gas analyzing equipment. Initial impurity concentration prior to the purification showed approximately 50 ppm water moisture and 30 ppm oxygen. Since the residual gas analyzer was qualitative, only relative amounts of other residuals should be obtained. Once the original values were established, the purification heaters containing the shredded titanium foil were energized. Gas temperatures to the titanium foil area were maintained at  $800^{\circ}\text{F}$  in one gettering tank and at  $1500^{\circ}\text{F}$  in the other. Periodic checks with the analyzing equipment indicate a substantial lowering of the impurity level after the first 48 hours of operation. However, in a total of 17 days of purification operation, the oxygen content reached a value of 2 ppm  $\text{O}_2$ . Other trace impurities were also considered negligible.



SAMPLE CALIBRATION - SPUR TWO-PHASE  
VERTICAL LIQUID LEVEL PROBE

FIGURE 25

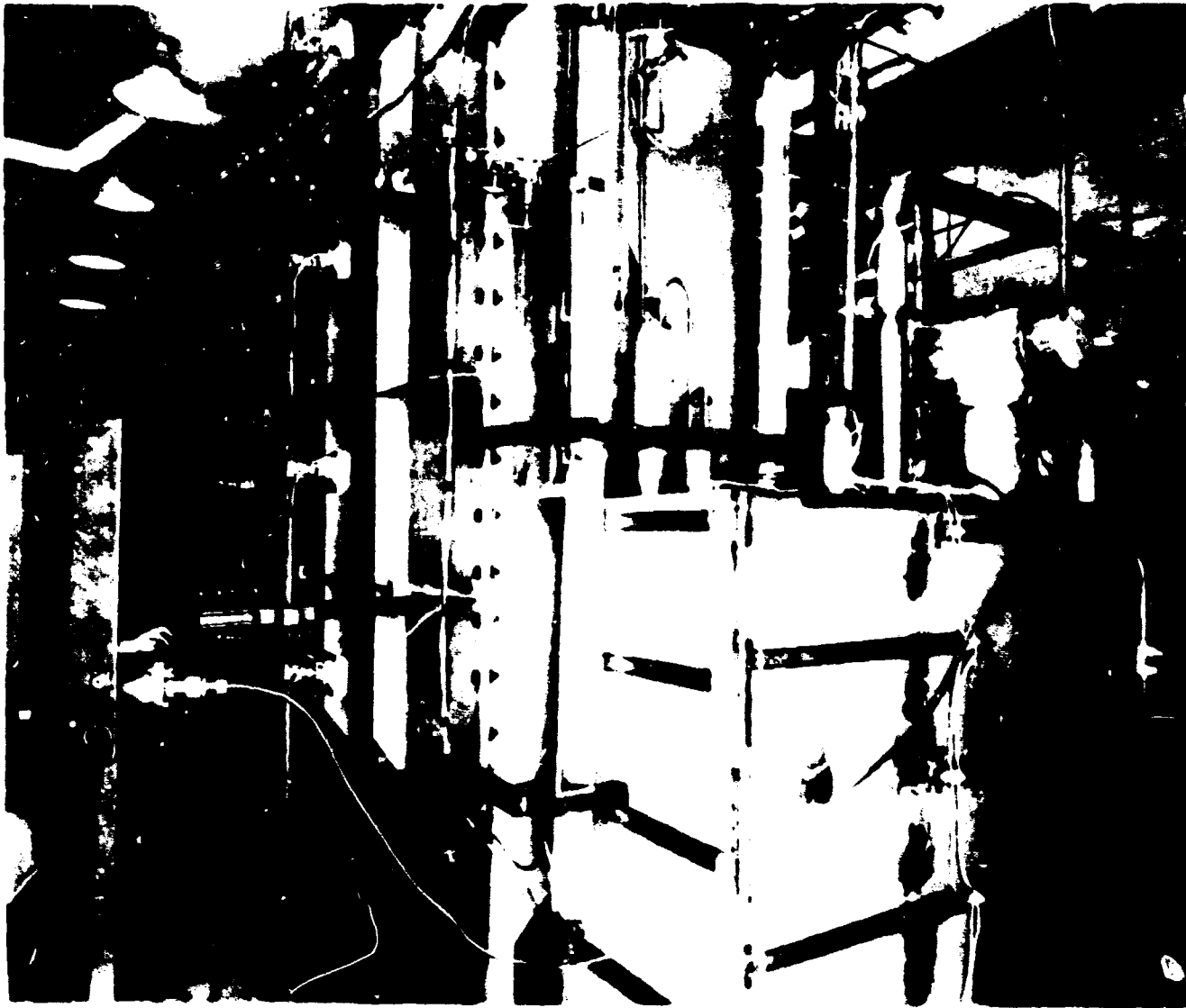


FIGURE 26. EXTERNAL HEATERS FOR CHAMBER BAKEOUT

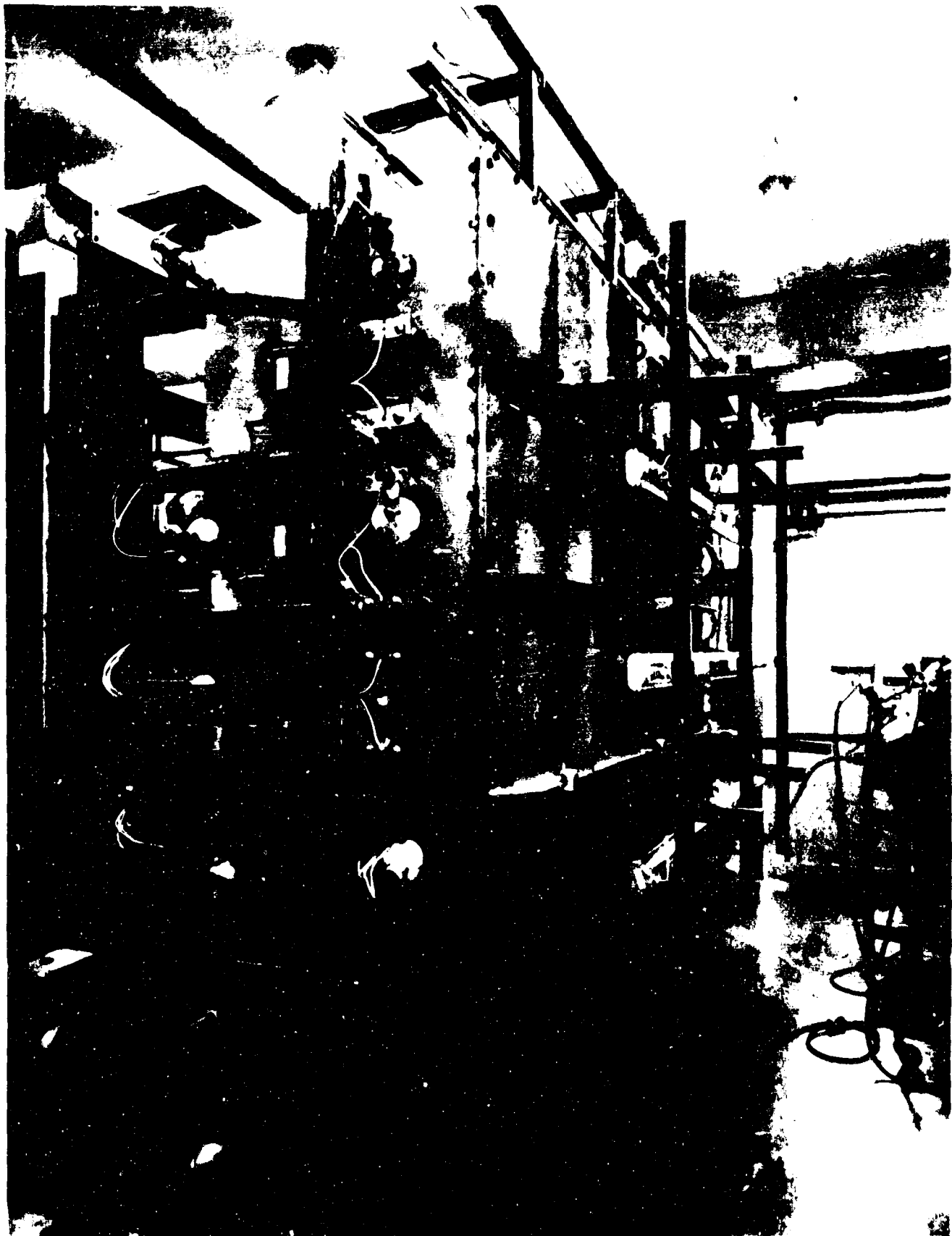


FIGURE 27. EXTERNAL HEATERS FOR CHAMBER BAKEOUT

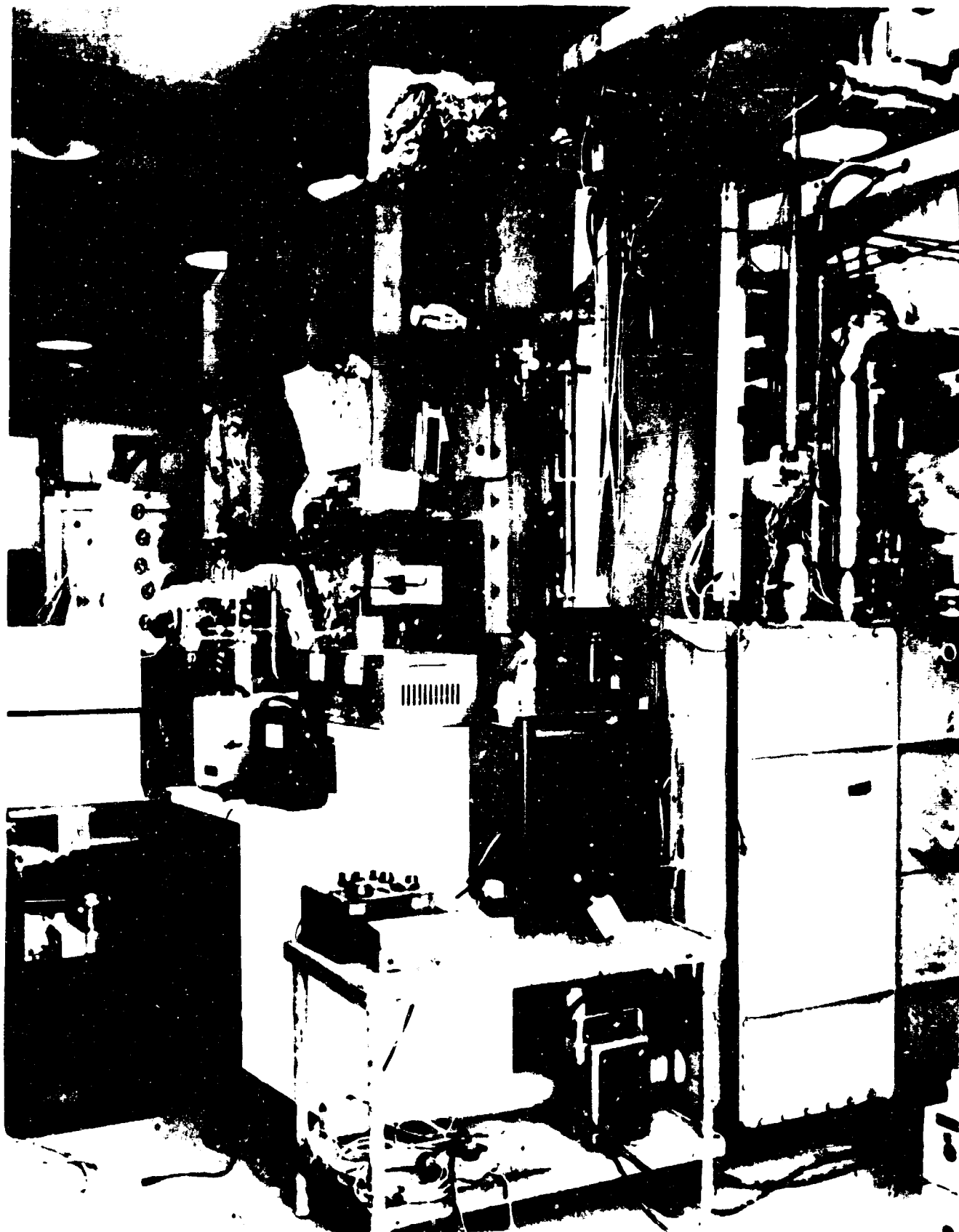


FIGURE 28. INERT-GAS IMPURITY MONITORING SYSTEM

As soon as this impurity level was reached, the annealing heaters which were placed directly over each field weld were energized. The temperature read by thermocouples placed on the tube adjacent to the weldments was 2200°F, and this temperature was held for 1 hour. A check of the argon purity during the annealing cycle revealed no increase in the possible contaminants.

After the annealing cycle was completed, potassium was transferred from the potassium shipper to the loop transfer pot containing zirconium sheet as shown in Figure 29. The potassium was gettered for 14 hours at 1400°F.

Upon completion of the potassium gettering cycle, the loop heaters were turned on to give loop temperatures of 400°F. The loop was then filled slowly with potassium to a predetermined level in the accumulator tank. As a result, the bottom portion of the loop was filled with liquid potassium, with the top horizontal and the major amount of both vertical legs of the loop having a vacuum of  $10^{-3}$  torr or better. A fill sample for chemical analysis was taken during the filling of the loop.

As soon as the fill was accomplished, the accumulator was pressurized to 10 psig and the loop proper was completely full of liquid potassium at a temperature of 400°F.

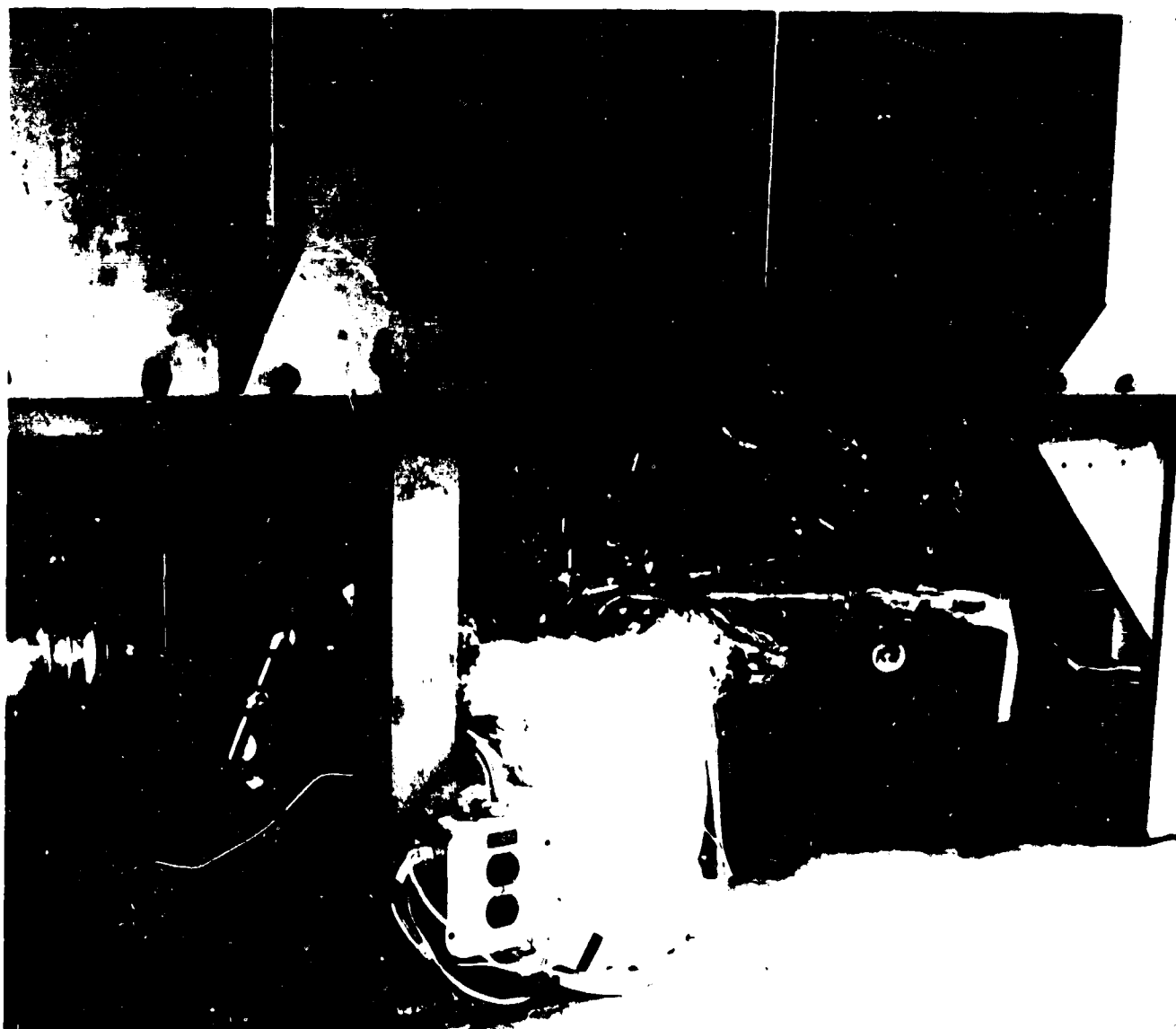


FIGURE 29. POTASSIUM TRANSFER SYSTEM



## SECTION VII LOOP OPERATION

The operation of the loop as shown in Table 1 included an isothermal 600°F all-liquid operation and a 4-hour test under boiling conditions. The goal of 1 000 hours under boiling conditions was not met. The purpose of the isothermal run was primarily to check out the instrumentation under flowing-potassium conditions. During this 72 hour nonboiling run, the flow rate was established at 0.25 pound per minute with the loop temperatures at approximately 600°F. A complete set of detailed operating procedures for the loop are listed in Appendix II.

TABLE 1  
LOOP OPERATION

Liquid isothermal run (600°F)	72 hours
Nonisothermal liquid run (to boiling)	24 hours
Boiling operation	307 hours

After the loop was operated isothermally at 600°F, power to the superheater was increased until boiling was established at the test-section exit. The condensing temperature was set at 1760°F by adjusting the accumulator argon cover pressure.

Boiling was increased by gradually increasing power input to the superheater section. The potassium flow rate was held constant by increasing the pump voltage to overcome the increasing loop pressure drop attendant with increasing vapor quality.

The boiling interface was shifted from the throttling section to the superheating section by gradually increasing boiler and superheater power. Passage of the boiling interface was verified by observing a gradual increase in liquid potassium temperature at a point followed by a gradual increase in two-phase potassium temperature as vapor generated upstream caused a drop in pressure at the point of observation. This procedure was continued until the boiling interface had been shifted to the middle section of the boiler.

The quality of the vapor generated was increased by increasing the power to the two-phase region until a small degree of superheat was achieved at the exit of the superheater. The superheated condition was verified by again observing the history of the superheater exit temperature which displayed a change from a gradual decrease with increasing boiler power (during two phase operation) to a gradual increase with increasing boiler power (during superheating operation).

Typical loop operating conditions are shown in Figure 30. Conditions are shown from preheater entrance to superheat exit. The condensing temperature was approximately 1760°F. This temperature was higher than originally intended 1500°F because the test-section (the orifice section containing the candidate turbine material) pressure drop was less than the anticipated value. In addition, a subcooled temperature of 212°F was experienced as a result of an oversized condenser section and the cooling-argon flow rate through this condenser section was in excess of the minimum controllable value. This was due to the fact that the argon feed to the condenser was in parallel to the argon coolant to the pump and the controlling valve was in the main argon line. If the argon flow rate was reduced, subsequent reduction of argon coolant to the pump and flowmeter would result. With the pump running at the maximum operating temperature, the coolant flow could not be reduced. The following average test conditions were observed during the test:

Liquid flow rate	0.6 pound per minute
Boiling temperature	1950°F
Vapor velocity:	
Orifice No. 1	Mach 0.8
Orifice No. 2	Mach 0.8
Orifice No. 3	Mach 0.8
Vapor temperature (at test section)	2000°F
Condenser inlet temperature	1760°F
Condenser outlet temperature	212°F
Heater inlet temperature	745°F

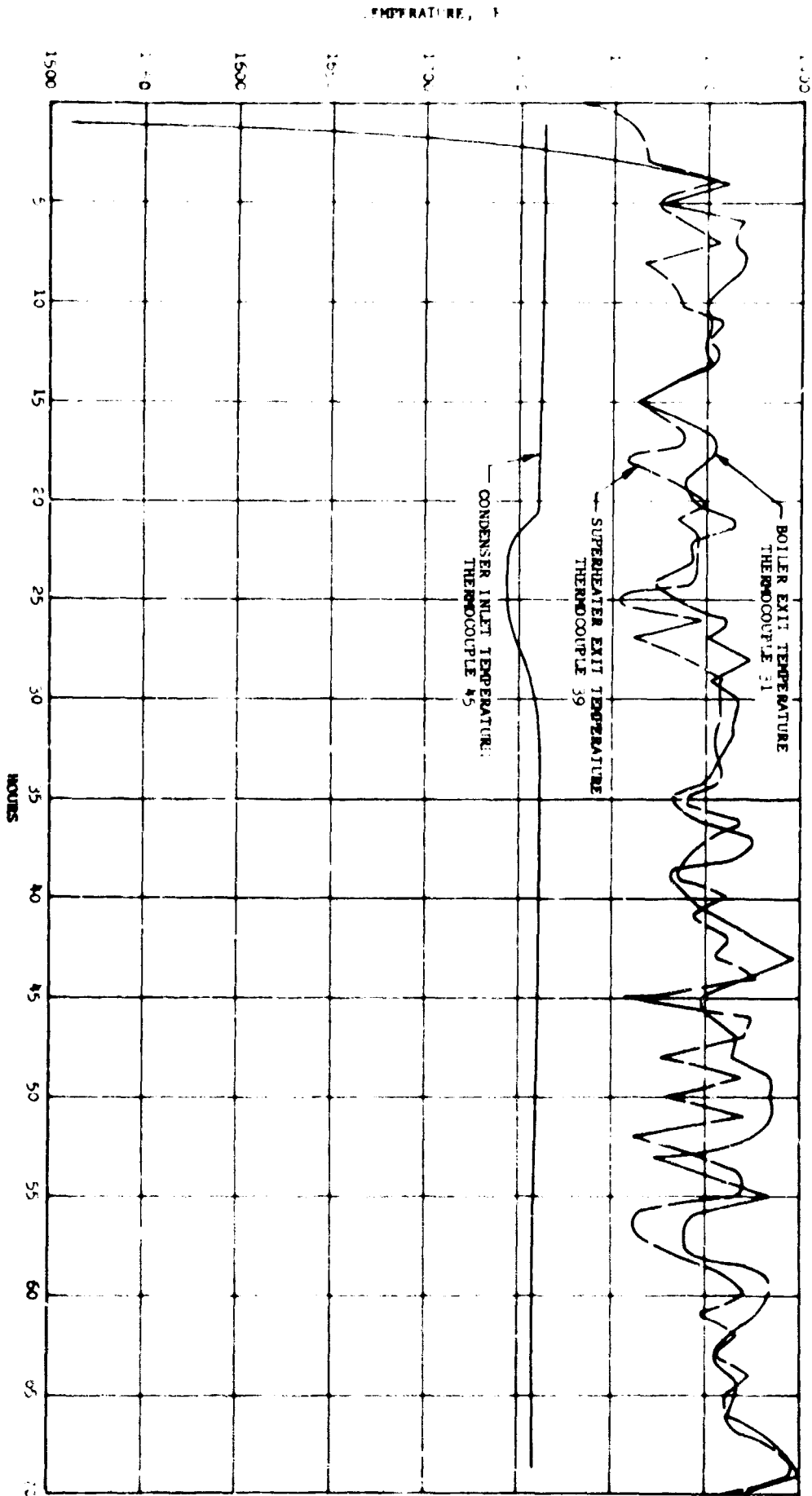


FIGURE 30. LOOP BOILING AND CONDENSING TEMPERATURES DURING INITIAL LOOP OPERATION

The maximum potassium flow rate was limited during the test by the liquid-metal pump temperature. Therefore a constant pump voltage was necessarily applied throughout the duration of the test. Figure 31 shows the gradual decrease in flow rate experienced at a constant pump voltage. While conclusive evidence is not available, the decrease in flow rate is believed to be the result of a gradual degradation of the twisted tape insert in the boiling and preheating sections.

During the boiling operation, the electromagnetic pump was operated at the maximum allowable flow rate as established by the pump winding temperature. A temperature of 410°F was experienced on the field windings, even though the pump was not operating at maximum input voltage. Although argon cooling for these windings was provided the amount was insufficient.

Periodic moisture, mass spectrographs, and oxygen analyses of the test argon were made during the test operation. Less than 1 ppm of oxygen and 10 ppm of water vapor were maintained throughout the test. Comparison of the traces of elements from mass numbers 0 to 80 prior to test and those during test revealed no change due to the high-temperature operation.

The boiling operation was terminated after 307 hours of testing when the flow dropped to almost zero. The low-flow alarm cut the power to the loop and the loop temperatures were reduced which allowed the loop potassium to become single-phase (liquid). Some flow in the all-liquid state was achieved; however, there was a severe reduction in pump efficiency, as noted by the pump input voltage. Therefore, a low-temperature (600°F liquid operation was pursued. Pump performance (flow rate versus input voltage) gradually decreased during this run. Resistance checks were made on the pump windings and revealed no degradation of the electrical insulation of the winding. After 12 hours of running time had elapsed, the flow stopped completely. By reversing the flow direction, flow was resumed. Again 4 hours later the flow was interrupted and the above process was repeated. After the third flow interruption, attempts were made to drain the loop and fill it with a fresh charge of potassium. Draining the loop was difficult, and a line restriction in the fill line was indicated. Despite these difficulties, the loop was refilled with potassium, and attempts to establish flow were made but were unsuccessful. Consequently, further loop operation was terminated. A drain sample for chemical analysis was taken during the first loop draining.

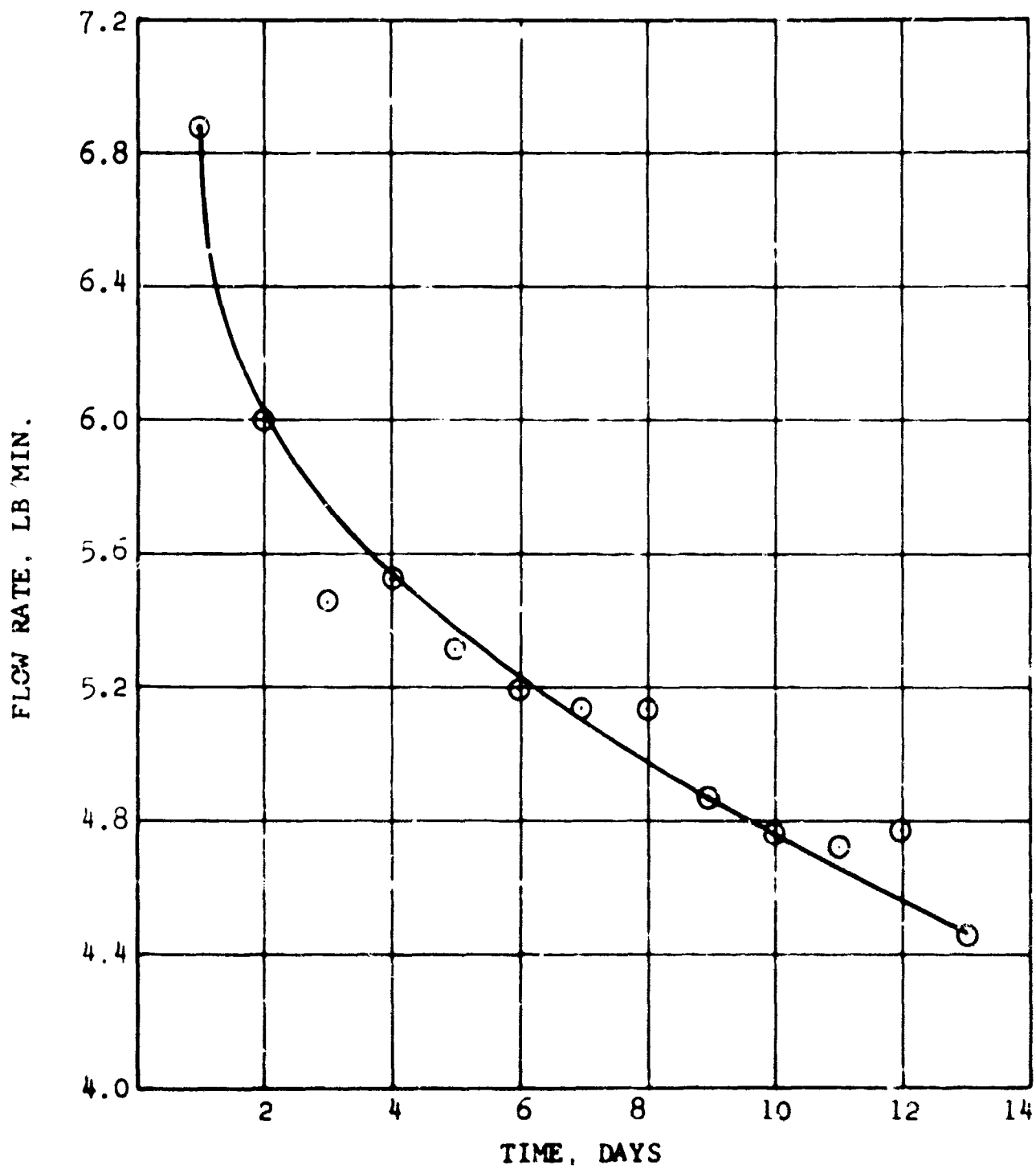


FIGURE 31. MASS FLOW RATE VERSUS OPERATIONAL TIME  
(PUMP VOLTAGE CONSTANT)

## SECTION VIII TEST RESULTS

After the loop and chamber had cooled to ambient room temperature the test chamber door was removed. Figures 32 and 33 show the loop and the various loop components after test. Meticulous systematic loop disassembly procedures were formulated and followed. Visual examination of the loop indicated areas of contamination. The boron nitride heater insulators were darkened, as shown in Figure 34, and the radiant shields showed a dark deposit, as indicated in Figure 35.

The thermal insulation and radiant heat shields were removed. Figure 36 shows the test loop with loop heaters intact but with the annealing heaters removed. A typical view of a disassembled annealing heater is shown in Figure 37.

The disassembled loop was radiographed prior to sectioning to determine the cause for flow failure. Upon completion of the x-ray examination, the loop was sectioned for metallography and chemical analysis. From the loop radiographs it was discovered that the twisted ribbon in the boiler section had deteriorated in two places approximately 10 inches apart, as shown in Figure 38. A 3-inch section of the ribbon was missing and found broken up in the one break and another piece, 6 1/2 inches in length, was loose in the boiler tubing. The loop was sectioned and the tube cut longitudinally to show the failure. Figure 39 shows the first break and the pieces of the  $\text{Cb} - 1.0 \text{ w/o Zr}$  twisted ribbon jammed against the ribbon that was still intact. This condition resulted in a flow restriction. Figure 40 shows the second break in the ribbon which occurred approximately 10 inches from the first break. Figure 41 shows both of the breaks in the turbulator and their relative positions in the boiler tubing.

Examination of the tubing wall where the ribbon failure had occurred showed excessive gouging from the loose turbulator. The area in which the ribbon actually broke off into small pieces shows circumferential grooves or gouges on the inside tubing wall. In the areas where the ribbon was loose at one end and fastened at the other, spiral grooves or gouges corresponding to the shape of the twisted ribbon were observed on the tube walls (see Figure 41). The rifling (spiral grooves) were present in the vapor region of the boiler and the region of the first two superheaters. No wall damage (grooves) was found in the preheater section, boiler inlet section, or superheater exit section.

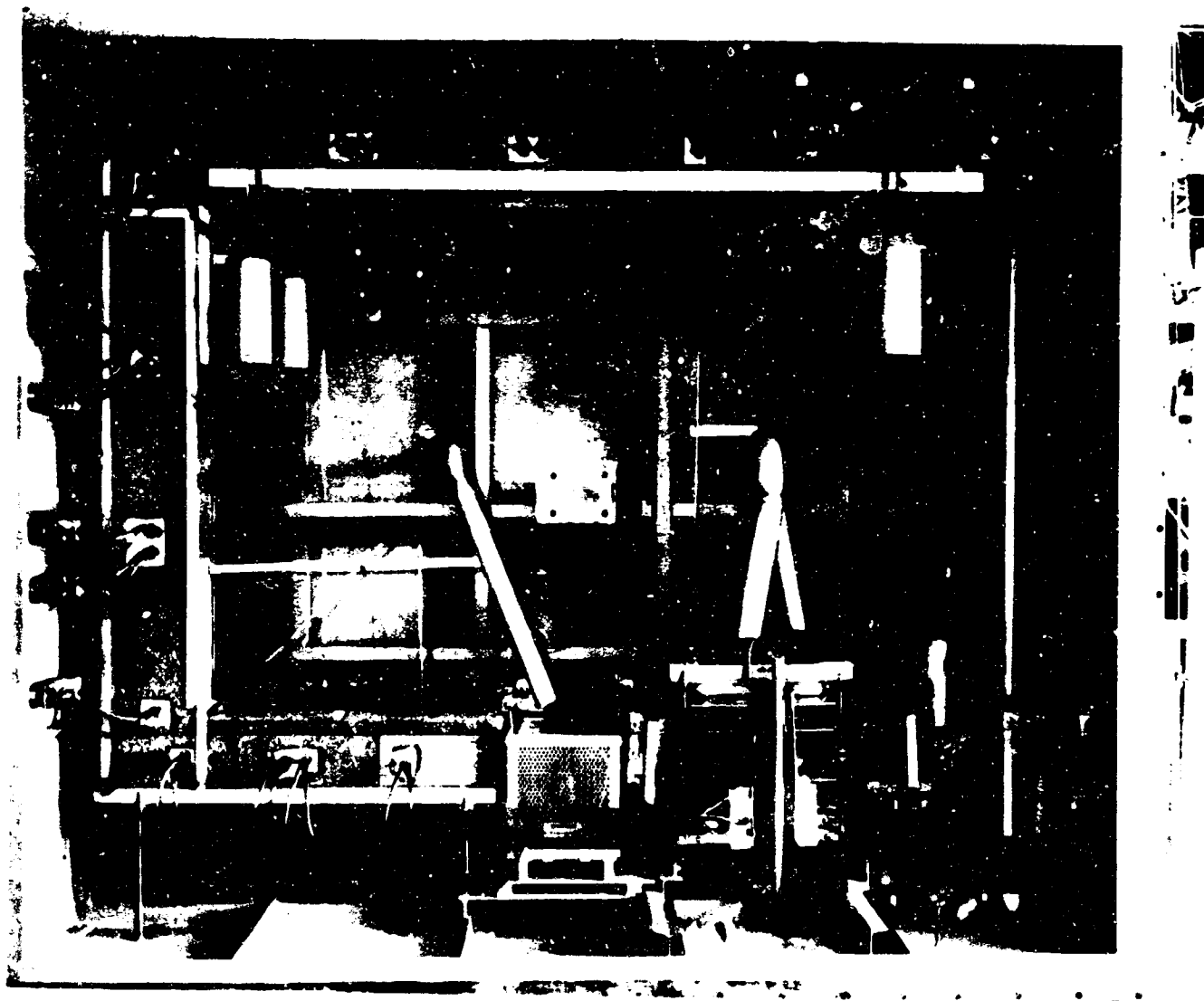


FIGURE 32. TEST LOOP IN CHAMBER AFTER TEST

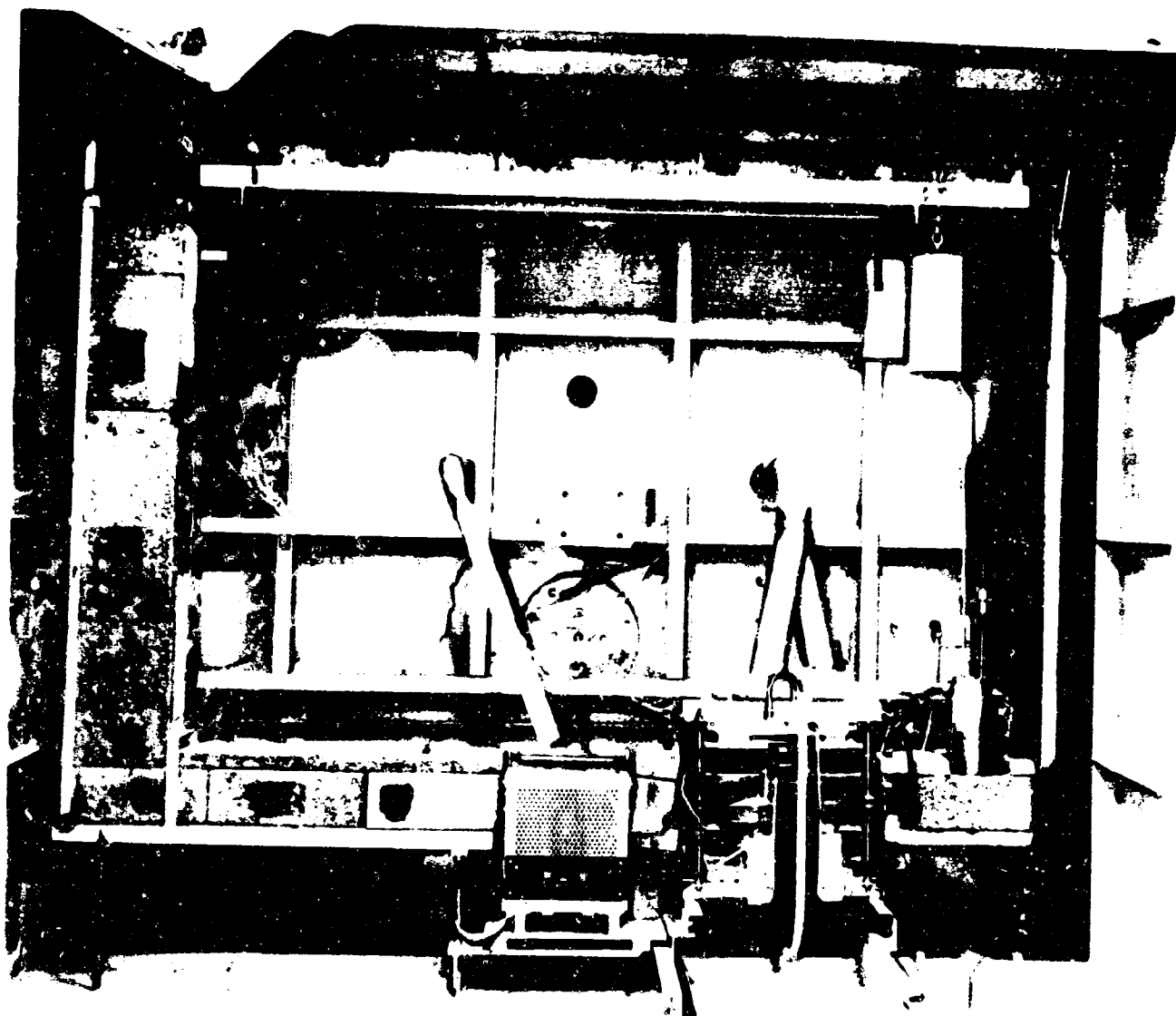


FIGURE 33. TEST LOOP IN CHAMBER AFTER TEST



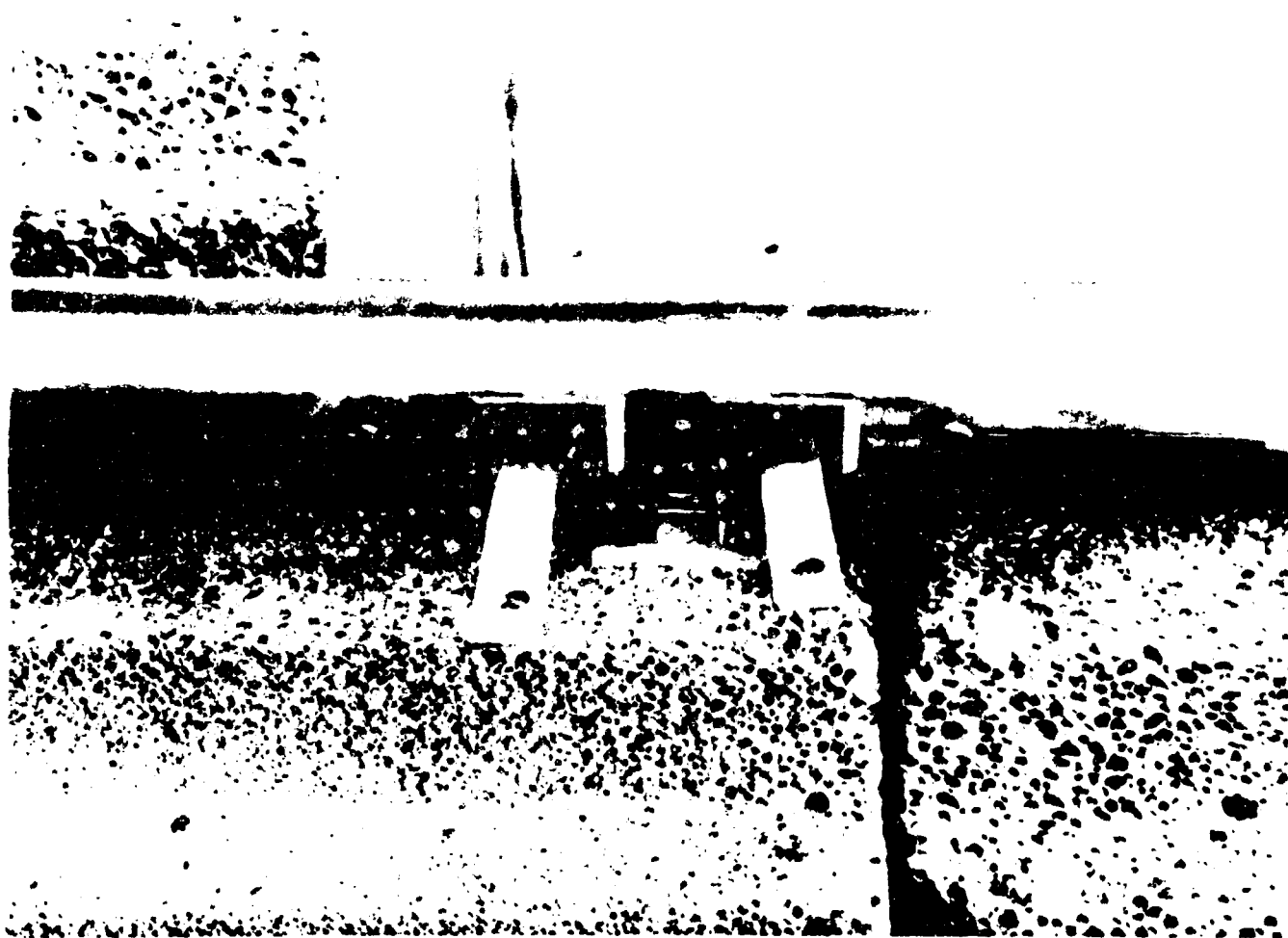


FIGURE 34. LOOP PREHEATER SECTION AFTER TEST



FIGURE 35. BOILER HEATER SECTION AFTER TEST



FIGURE 36. TWO-PHASE LOOP WITH THERMAL INSULATION  
AND RADIANT SHIELDS REMOVED (AFTER TEST)

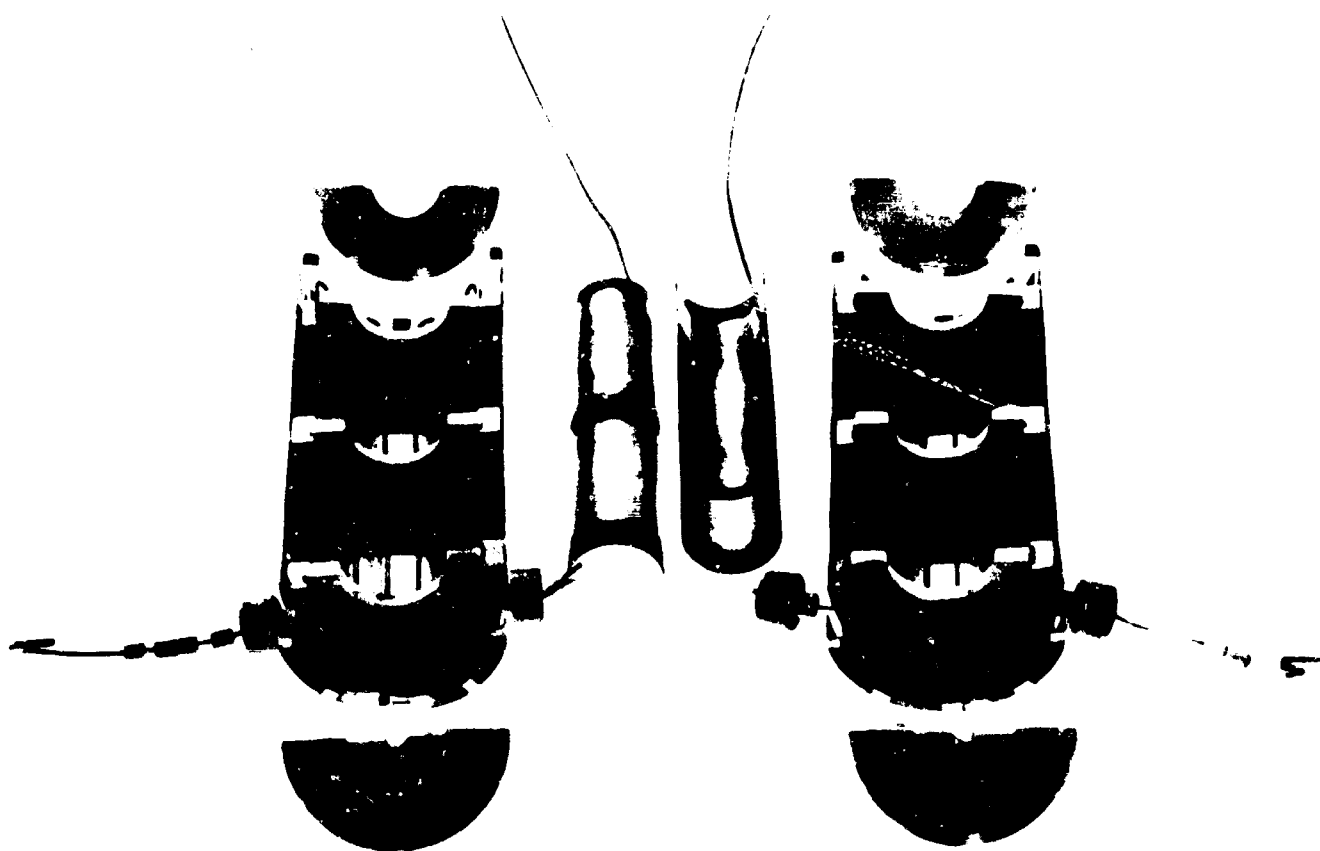
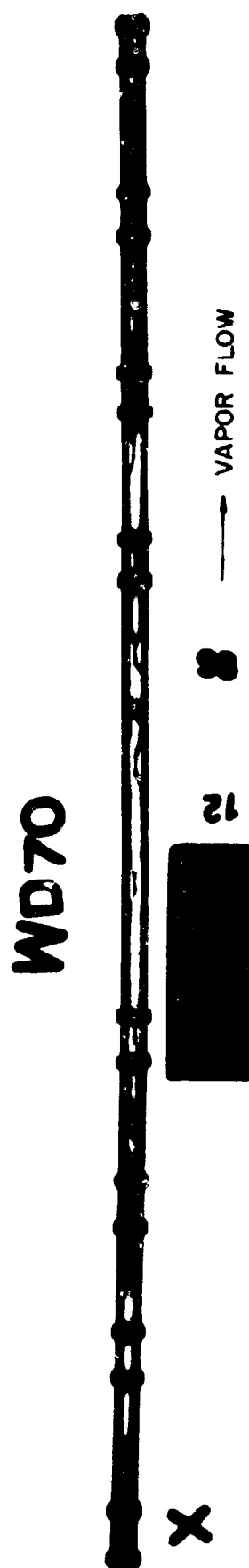


FIGURE 37. DISASSEMBLED ANNEALING HEATER AFTER TEST



SCALE: 1 INCH 2 1/2 INCHES

FIGURE 38. RADIOGRAPH OF HEATER SECTION WHERE RIBBON FAILURE OCCURRED



FIGURE 39. LOOP TUBING CUT LONGITUDINALLY WHICH  
SHOWS THE RIBBON FAILURE MAGNIFICATION 2.8X

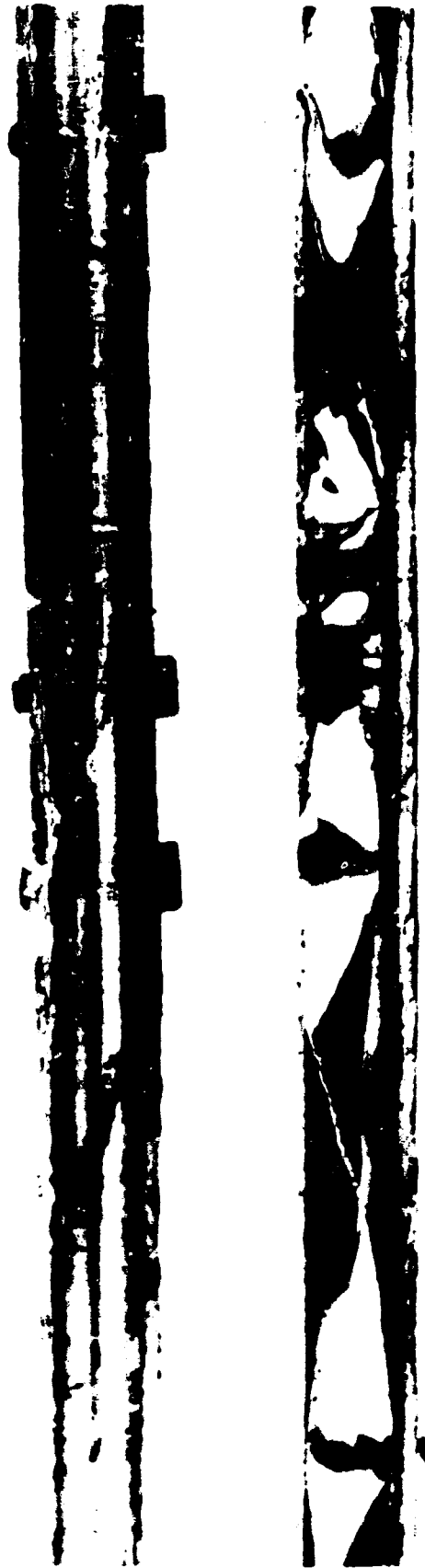
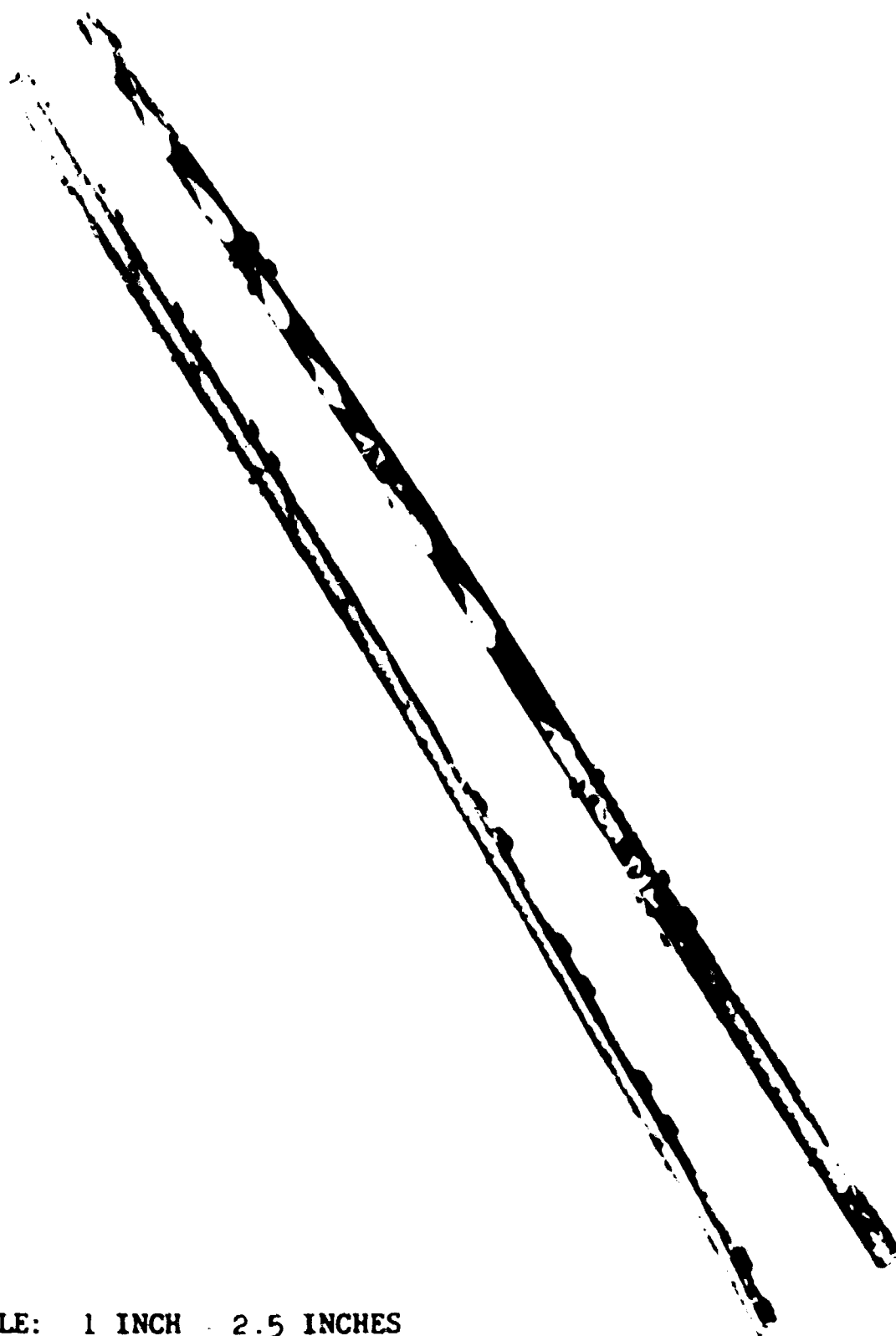


FIGURE 40. SECOND RIBBON FAILURE LOCATED IN THE BOILER SECTION  
MAGNIFICATION 2.8X



SCALE: 1 INCH . 2.5 INCHES

FIGURE 41. LONGITUDINAL VIEW OF BOILER SECTION  
SHOWING THE RIBBON FAILURES



Examination of the ribbon in both the failure area and the vapor regions of the heater sections showed excessive wear where the ribbon had contacted the tubing wall. Figure 42 shows the ribbon damage in the failure area. The type of damage to the ribbon in the superheater section (the liquid region) showed the absence of any form of damage (see Figure 43).

The test section containing the fixed orifices and the test coupons of Mo + 0.5 w/o Ti was removed from the loop and decontaminated. In attempting to remove orifice No. 3 from the test-section housing, the front surface of the orifice was scratched. Figure 44 shows the orifices and the test coupons prior to final decontamination. The discolorations of the pieces occurred during the preliminary decontamination. Macro-examination of the test coupon surfaces (Figures 45, 46, and 47) showed evidence of vapor impingement. The surfaces prior to test were polished to a mirror finish; and due to the difference in reflectivity of the surfaces after test, the vapor impingement area was visibly noticeable. After final potassium decontamination, the dimensions of the orifices and the test coupons were measured. Orifice No. 1, which was fabricated from Mo + 0.5 w/o Ti and had an orifice diameter of 0.0765 inch, showed no change in diameter as measured with a Pratt and Whitney Supermicrometer. Orifices No. 2 and No. 3, which were fabricated from Cb + 1.0 w/o Zr and had initial orifice diameters of 0.0943 and 0.1196 inch, respectively, showed a diameter increase of 0.0003 and 0.0004 inch. The orifice diameters were sized to give near sonic (Mach 0.8) vapor velocities under the test conditions. Thickness measurements of the Mo + 0.5 w/o Ti test coupons showed no measurable thickness changes. The weight change measurements of the coupons were as follows:

<u>Test Coupon No</u>	<u>Original Wt. gms</u>	<u>After Test Wt. gms</u>	<u>Weight Change. gms</u>
1	2.7083	2.7081	-0.0002
2	2.7032	2.7024	0.0008
3	2.7219	2.7217	-0.0002

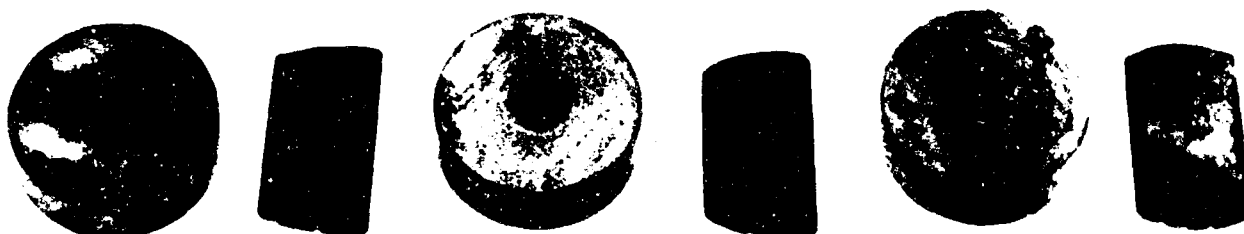
Metallographic examination of a section across the impingement area of the test coupons showed no surface irregularities, as indicated by Figures 48, 49, and 50. A typical photomicrograph of an orifice cross section is shown in Figure 51. This also displays no apparent surface degradation due to the high-velocity vapor.



FIGURE 42. DETAILS OF THE RIBBON LOCATED NEAR THE FLOW-RESTRICTION AREA - MAGNIFICATION 7X



FIGURE 43. RIBBON TAKEN FROM PREHEATER SECTION OF  
LOOP AFTER TEST MAGNIFICATION - 7X



SCALE: 1 INCH = 1.5 INCHES

FIGURE 44. TEST ORIFICES AND TEST SECTIONS  
AS REMOVED FROM DECONTAMINATED LOOP



FIGURE 45. MACRO OF VAPOR IMPINGEMENT AREA  
ON TEST SPECIMEN NO. 1  
MAGNIFICATION - 100X



FIGURE 46. MACRO OF VAPOR IMPINGEMENT AREA  
ON TEST SPECIMEN NO. 2  
MAGNIFICATION - 100X

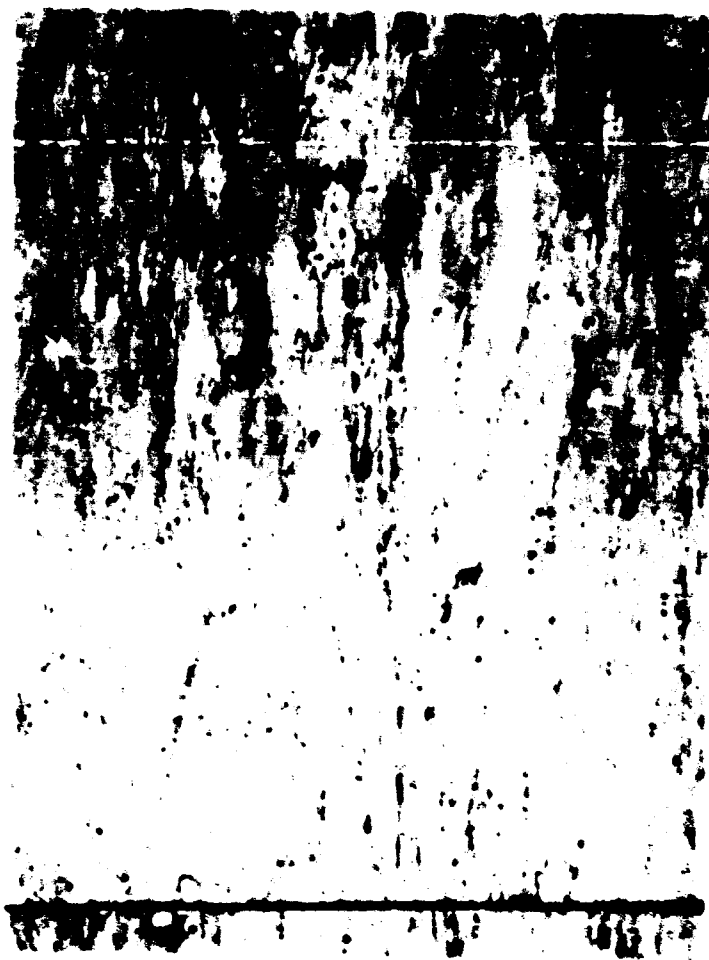


FIGURE 47. MACRO OF VAPOR IMPINGEMENT AREA  
ON TEST SPECIMEN NO. 3  
MAGNIFICATION - 100X

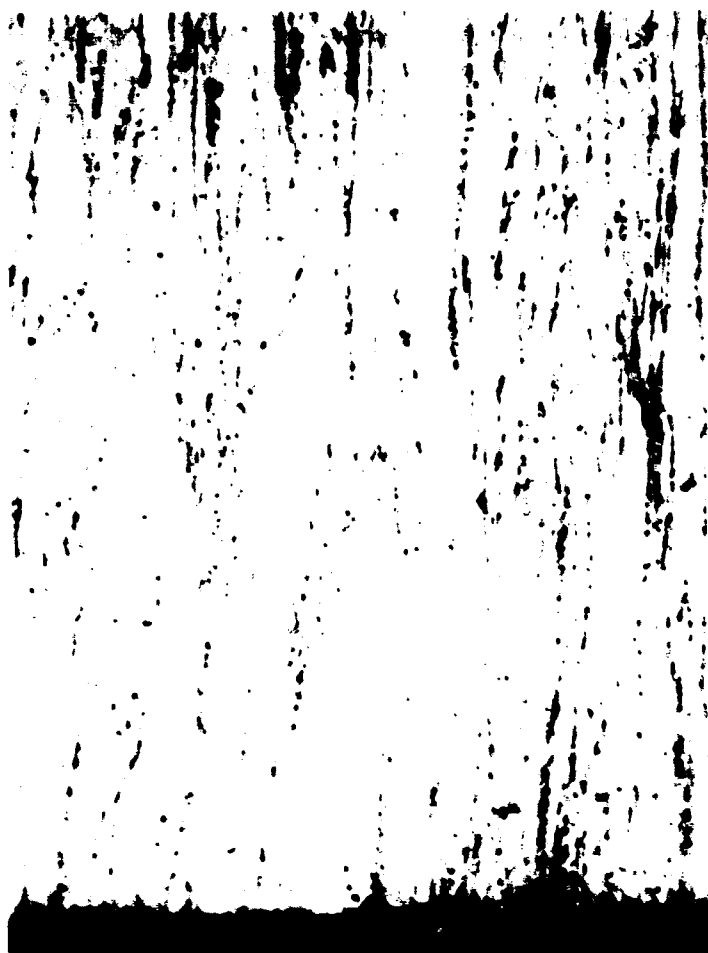


**FIGURE 48. PHOTOMICROGRAPH (200X) OF TEST SPECIMEN NO. 1  
SHOWING A VIEW TAKEN THROUGH THE VAPOR  
IMPINGEMENT AREA**





**FIGURE 49. PHOTOMICROGRAPH (200X) OF TEST SPECIMEN NO. 2  
SHOWING A VIEW TAKEN THROUGH THE VAPOR  
IMPINGEMENT AREA**



**FIGURE 50. PHOTOMICROGRAPH (200X) OF TEST SPECIMEN NO. 3  
SHOWING A VIEW TAKEN THROUGH THE VAPOR  
IMPINGEMENT AREA**



FIGURE 51. PHOTOMICROGRAPH (200X) OF TEST ORIFICE NO. 1  
SHOWING THE ORIFICE SECTION THAT WAS EXPOSED  
TO HIGH-VELOCITY POTASSIUM VAPOR

Metallographic and chemical samples were sectioned from the loop according to Figure 52. The temperatures which the samples experienced for the 307 hours of boiling are as follows:

<u>Metallography Samples</u>	<u>Temperature, °F</u>
<u>Control</u>	
1 tubing	770
2 tubing	1000
3 tubing	1350
4 tubing	1900
5 tubing	1930
6 tubing	1930
7 tubing plus weld	1930

Test Section

8 tubing	1779
----------	------

<u>Chemistry Samples</u>	<u>Temperature, °F</u>
1	1900
2	1930
3	1930
4	212

The chemical analysis of the various loop materials after test included analyses on the following samples:

- (1) Tubing material which was taken in layers from the tubing OD: first cut - 0.002 inch; second cut - 0.0005 inch; third cut - 0.005 inch; and the remainder of the tube wall - 0.036 inch.
- (2) A bulk tubing sample.
- (3) A bulk sample from the 0.003-inch thick tantalum wrapping on the boiler section

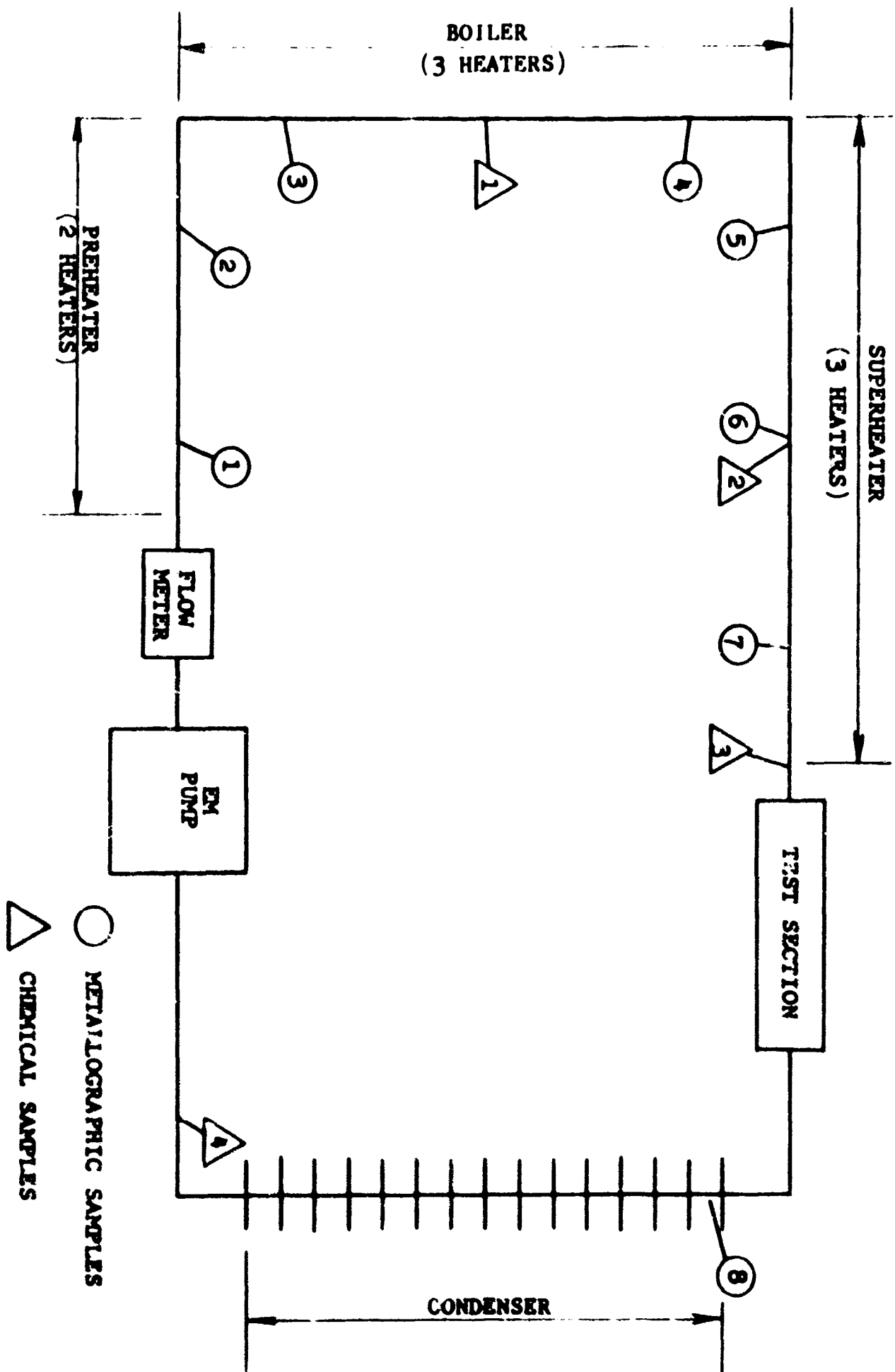


FIGURE 52. SAMPLE POSITIONS ON LOOP

- (4) A black deposit found on the tantalum radiant heaters.
- (5) A bulk sample from the tantalum radiant shield in the boiler section.
- (6) A dark layer on the boron nitride insulators used in the heater section.
- (7) A sample of Foamsil taken from the boiler section.
- (8) Potassium samples taken before and after test.

The metal analysis included the analysis for the interstitial impurities,  $O_2$ ,  $N_2$ , and  $H_2$ . The analyses were performed by the vacuum fusion method of a Meco Vacuum Fusion Analyzer. The deposit and layer formation were analyzed by an ARL Emission Spectrograph. The potassium analysis was performed by the mercury amalgamation process. During the potassium sample batching down process, which is required to prepare a sample for oxygen analysis, it was found that the drain sample contained no potassium. Since the draining of the loop was difficult, there was no positive means of knowing if the sample tube was filled during the draining of the loop. As a result, the chemical analysis of the potassium after test is not available. The following analysis was obtained on the fill sample: 35  $O_2$ , 20 Fe, 20 Si, <50 Mn <10 Mg, <50 Cr, <10 Sn, 10 Ni, 10 Al, <10 Mo; <10 V. <10 Cu. <10 Ag, <10 Ti, and 20 Ca (all figures in parts per million by weight).

Tables 2 and 3 show the chemical results for the interstitial content of the Cb + 1.0 w/o Zr and the Ta, respectively, before and after test. The black deposit found on the tantalum radiant heaters was found to be principally a carbonaceous compound. The layer found on the boron nitride insulators was also found to be carbonaceous. This layer was also found by a crude electrical measurement to be slightly electrically conductive. The glazed area found on the Foamsil in the boiler section adjacent to the radiant shield showed the presence of 0.1 w/o Fe.

The metallography included hardness traverses across the tube wall as well as microstructure analyses. Metallography was performed with the use of a Leitz Research Metallograph. Standard polishing equipment such as a Zeiss Electropolisher, vibrating automatic polishing laps, etc. was used in the sample preparation. Hardness measurements were taken by a Wilson Tukon Microhardness Tester. The etchant used for the metallography was as follows: 20 grams  $NH_4F$  HF, 50 cu cm  $HNO_3$ , and 100 cu cm  $H_2O$ .

TABLE 2  
VACUUM FUSION ANALYSES OF Cb + 1.0 w/o Zr LOOP MATERIAL

		O <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub>
<u>Pretest Analyses</u>		(ppm by weight)		
A.	Cb + 1.0 w/o Zr Tubing, as received	300	24	5
<u>Post-Test Analyses</u>				
A.	Cb + 1.0 w/o Zr Tubing, Sample 1			
	1. 1st 0.003-inch layer, OD	2860	334	43
	2. 2nd 0.005-inch layer, OD	2290	210	39
	3. 3rd 0.005-inch layer, OD	2260	210	39
	4. Remainder of tube wall	1285	167	51
B.	Cb + 1.0 w/o Zr Tubing, Sample 2			
	1. 1st 0.003-inch layer, OD	2104	210	47
	2. 2nd 0.005-inch layer, OD	1210	210	43
	3. 3rd 0.005-inch layer, OD	775	210	35
	4. Remainder of tube wall	667	100	44
C.	Cb + 1.0 w/o Zr Tubing, Sample 3			
	1. 1st 0.003-inch layer, OD	2570	210	51
	2. 2nd 0.005-inch layer, OD	2080	146	58
	3. 3rd 0.005-inch layer, OD	1941	145	24
	4. Remainder of tube wall	800	129	48
D.	Cb + 1.0 w/o Zr Tubing, Bulk Sample 4	430	55	7

TABLE 3  
VACUUM FUSION ANALYSES OF TANTALUM MATERIAL  
BEFORE AND AFTER TEST

	Oxygen		Nitrogen		Hydrogen	
			(ppm by weight)			
	Before	After	Before	After	Before	After
1. 5/8-inch-OD tantalum heater, boiler section	34	3015	41	1174	<10	<10
2. 0.003-inch- thick tantalum wrapping boiler section	26	1580	24	20	<10	29
3. Inner tantalum radiant shield, boiler section	30	488	20	17	<10	<10



The hardness measurements are shown in Table 4. Again, many samples were run; however, these values are representative of the values found in the various portions of the loop.

The following metallography was performed on the loop tubing. (The photomicrographs are shown in Appendix I.) The operating temperature accompanies the sample identification number and the magnification.

Control Sample (200X). The microstructure is normal for annealed rolled material. One fabrication defect for each 0.002 inch was observed.

1 Preheater (770°F, 200X). No surface roughness due to the test fluid was observed. The recrystallized structure is similar to that found in the as-received (control) sample.

2 Preheater (100°F, 200X) Similar to the above pre-heater sample.

3 Boiler (1350°F, 200X). Similar to preheater samples 1 and 2.

4 Boiler (1900°F, 200X) A noticeable metalloid phase was observed throughout the structure, which indicated a structure typical of contaminated Cb + 1.0 w/o Zr. Surface smoothness is the same as in the as-received condition.

5 Superheater (1930°F, 200X). Similar to sample 4; however there was a substantial increase in the dark salt- and pepper phase which indicates excessive interstitial contamination.

6 Superheater (1930°F, 200X). Similar to sample 4; however from general appearance, the metalloid phase exists in greater amounts.

7 Superheater Outlet (1930°F, 50X) This is a longitudinal section of a weldment--a distinct white band is visible near the ID of the tube, which is void of the metalloid phase. This is typical of contaminated Cb + 1.0 w/o Zr that is annealed. It is believed that this zone contains a greater oxygen content in solution. The sample was overetched to bring out the structure. A transverse section including the entire wall thickness shows a contaminated structure throughout the wall thickness. Both specimens appear to be in a solution-treated and aged condition.

8 Condenser Outlet (212°F, 200X). This section has the typical microstructure of annealed rolled material. Surface condition is the same as in the as-received condition. The dark metalloid phase that was in some of the other specimens is absent.

TABLE 4  
MICROHARDNESS TRAVERSE ACROSS  
Cb + 1.0 w/o Zr TURBINE WALL

		Microhardness, Knoop (100-g Load)						
Sample		Distance from ID of Tubing, Inches x 10 <sup>3</sup>						
		6	12	18	24	30	36	42
1.	As-received tubing	155	131	135	135	141	135	140
2.	1 , Preheater	138	135	135	133	113	125	132
3.	2 , Preheater	125	115	112	110	116	124	152
4.	3 , Boiler	111	116	121	125	135	125	135
5.	4 , Boiler	108	112	104	112	126	132	124
6.	5 , Superheater	127	127	130	124	138	140	124
7.	6 , Superheater	112	118	110	110	113	116	128
8.	7 , Superheater	108	120	112	113	121	135	141
9.	8 , Condenser outlet	141	135	136	138	135	-	-

## SECTION IX - DISCUSSION OF RESULTS

The results can be summarized in five main divisions, namely: (1) the loop operation from a boiling standpoint, (2) the loop failure, (3) the test environment and the refractory-metal loop contamination, (4) potassium corrosion and/or erosion of loop materials, and (5) the peripheral test equipment.

The direct measurement of potassium vapor pressure in the boiling loop was not provided for in the loop design. This was largely due to the monometallic loop design restriction and the unavailability of reliable refractory metal (Cb - 10 w/o Zr) pressure-measuring devices.

To obtain vapor quality for the test, a heat balance was performed on the system during its boiling operation. Results obtained by this calculation do not yield absolute values, but do give an estimation of vapor quality. The following heat balance was determined from typical data taken during the boiling operation at design conditions:

Total flow rate	0.6 lb per min
Total power input	840 Btu per min
Heat losses	288 Btu per min
Sensible heat	129 Btu per min
Latent heat	423 Btu per min
Vapor flow rate	0.541 lb per min
Quality	90 percent

Heat losses in the preheater section were determined by total heat input minus sensible heat imparted to the potassium. It was assumed that heat losses from the boiler and superheater were the same per unit length as from the preheater. Whereas the actual heat loss should be greater in the boiler and superheater, unless some unknown influences were acting, it does appear that the ambient conditions for the superheater and boiler insulation were 400°F higher than the argon surrounding the preheater insulation.

The heat balance shows a quality at the superheater exit of 80 percent, while comparison of the temperature measured at the superheater exit to a temperature in a known two-phase region upstream indicates that the potassium exiting from the superheater was superheated. This strongly suggests a nonequilibrium mixture of superheated vapor and a liquid phase. The fluid stream probably contained liquid droplets and/or a stream of liquid flowing along the twisted ribbon insert.

During much of the operation the flow rate was fluctuating at greater than  $\pm 14$  percent. A 10-percent flow error would result in a 2-percent quality calculation error. A 20-percent error in the heat-leak determination would result in a 7-percent error in calculating quality. From these two possible sources of error the calculated quality could range from 80 percent to 100 percent. However, the pressure drop generated in the orificed test section indicates that the flow rate of vapor exiting from the superheater would not have exceeded 0.235 lb per minute or a quality of approximately 39 percent. Furthermore, a pressure balance around the loop substantiates the 39-percent figure. There is no apparent resolution of the discrepancy between the 80 percent calculated quality and the 39-percent indicated quality.

Unequivocally, the flow interruption that terminated the boiling operation and the subsequent plugging condition of the loop can be attributed to the failure of the twisted ribbon within the heater section. Since only the ends of the ribbons were attached to the tubing ID, the ribbon could conceivably have been subjected to a cyclic (vibratory) force which could have resulted in fatigue failure of the material. Although the liquid level in the boiler was not accurately known, it is anticipated that the ribbon failure occurred near the liquid-vapor region. Consequently, liquid-vapor interface perturbations during the boiling operation could have also contributed to the degradation of the twisted ribbon.

The test environment, although purified and maintained at low impurity levels, was not adequate for this test as manifested by the refractory-alloy contamination. Despite the extreme care exercised in the monitoring of the contaminant level, contamination of the loop materials, tantalum and the columbium alloy, resulted. The major contaminants, in descending order of magnitude, were oxygen, carbon, and nitrogen. The source of contamination was neither external to the test chamber nor external to its associated support equipment. The residual gas analyzer that was used as an impurity-monitoring device is extremely sensitive to external (air) leakage. Detection of external leakage by this instrument is categorically positive. Therefore, the introduction of contaminants to the test environment must have been from the test hardware itself.

Outgassing of clean metal surfaces exposed to vacuum conditions can be eliminated by a bakeout treatment at 300°F. Since this procedure was included in the test preparation, it can be assumed that the contribution to the contaminant level by this source was negligible. By a process of elimination, the thermal insulation, Foamsil, and the ceramic electrical insulation, boron nitride, remain as possible sources of contamination. It was found in earlier tests that these materials, in the as-received condition, can contaminate refractory alloys at elevated temperatures. Consequently, special procedures were established in bench-type tests to eliminate the contamination effects. However, despite these special treatments and the special handling during the incorporation of these materials on the loop, it is assumed that adsorption and absorption of impurity gases occurred after installation.

Apparently, the low-temperature (300°F) outgassing treatment prior to loop operation at elevated temperatures was not sufficient to eliminate the impurities. The contamination of the tubing, as evidenced by the chemical analysis, did not result in an increase in hardness. Also, the tubing material exhibited good ductility. This probably can be attributed to the solution treatment and overaging during exposure to the test temperatures.

Macro- and micro-examination of the loop revealed no evidences of attack by the potassium under the test conditions employed. Although the twisted ribbon in the areas where the ribbon had failed had eroded the tube wall from metal-to-metal contact, and had possibly precluded any tube-wall degradation due to corrosion effects in these areas, the condition of the remainder of the loop indicated no measurable corrosion. Careful dissection and potassium decontamination of the loop revealed no metallic particles deposits, or layers on the loop wall. In addition, the portion of the loop (the preheater section) that retained the potassium and was subsequently decontaminated indicated no metal particles. It was anticipated that because of the nature of the twisted-ribbon degradation, metal particles from the ribbon or adjacent tube wall would be transported by the liquid and/or vapor, causing damage to the test coupons and orifices. However, the results show very little, if any, change in the surface condition of the coupons and orifices, either due to high-velocity potassium vapor or to any metallic carryover (particles). Potential damage to the test coupons and the orifices was probably minimized by the ductile behavior of the twisted ribbon and tube-wall material.

The other support equipment, such as loop heaters, controls, instrumentation, and readout equipment, functioned satisfactorily during the test.

## SECTION X - CONCLUSIONS

The following conclusions are presented as a result of the test data obtained:

1. For the test conditions employed, there are no evidences of corrosion and/or erosion of the loop materials by the potassium liquid and/or vapor.
2. The loop heaters, both annealing and main heaters, were adequate for supplying the necessary heat input to the loop during test conditions.
3. Termination of the loop test was attributed to the degradation of the twisted ribbon that was inserted in the tubing for boiling stability.
4. Excessive contamination of the Cb - 1.0 w/o Zr loop material resulted from impurities in the test environment. This occurred despite the extreme precautions that were followed to eliminate these contaminants.
5. The source of contamination appears to be internal (outgassing) to the system rather than external.
6. Measurement of impurities in the ppm range in argon pressurized systems is extremely difficult.

## SECTION XI -RECOMMENDATIONS

The following recommendations are presented for future tests to determine dynamic potassium liquid and/or vapor corrosion and erosion behavior on refractory alloys at elevated temperature:

1. In order to obtain meaningful data that is directly applicable to power-conversion systems such as SNAP 50/SPUR, the test loop should be designed so that the operating conditions are similar to those of the power-conversion system, such as the loop that was employed in the above test. For example, the method of boiling, such as forced-circulation boiling, can greatly influence the corrosion behavior of boiler materials as well as the amount of carryover of boiler corrosion products by the vapor. Consequently, stagnant boiling such as pool-type boilers, could yield different corrosion characteristics. A boiler design for forced-circulation boiling can become quite complex; and as a result, stagnant boilers are frequently used in dynamic boiling corrosion tests. Extrapolation of corrosion data obtained by stagnant boiling to power-conversion systems employing forced-circulation boiling can be dangerous and misleading.
2. Special instrumentation development, such as pressure-measuring devices and vapor-quality measuring devices for refractory-alloy loops should be initiated.
3. Since it is extremely difficult to measure impurity level at the parts per million range in pressurized argon systems, other environments, such as ultra vacuums ( $10^{-5}$  torr or better) should be considered for tests in which the Cb - 10 w/o Zr is exposed to elevated temperatures.
4. The boron nitride used in contact with the refractory materials, tantalum or Cb - 10 w/o Zr, should be substituted with a denser ceramic such as alumina or beryllia. These materials are good electrical insulators and exhibit good thermal stability up to 2200°F with the refractory alloys.
5. Since this test operated for 307 hours, additional investigation for longer test times should be conducted.

## APPENDIX I

### METALLOGRAPHY OF Cb + 1.0 w/o Zr LOOP SAMPLES

Photomicrographs are of samples in the etched condition and the exposed edge is the tube wall that was exposed to dynamic potassium.

The etchant used was:

20 grams  $\text{NH}_4\text{FHF}$

50 cu cm  $\text{HNO}_3$

100 cu cm  $\text{H}_2\text{O}$



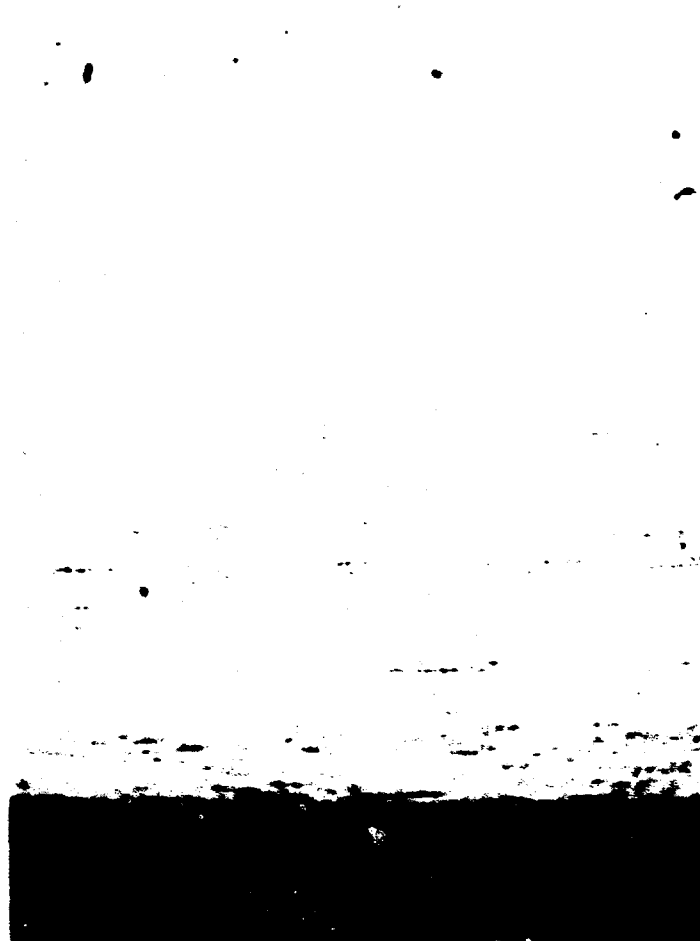


FIGURE 53. CONTROL SAMPLE: Cb + 1.0 w/o Zr, ANNEALED  
1 HOUR AT 2200°F, LONGITUDINAL SECTION, 200X



FIGURE 54. 1 PREHEATER, 200X, EXPOSURE  
TEMPERATURE 770°F

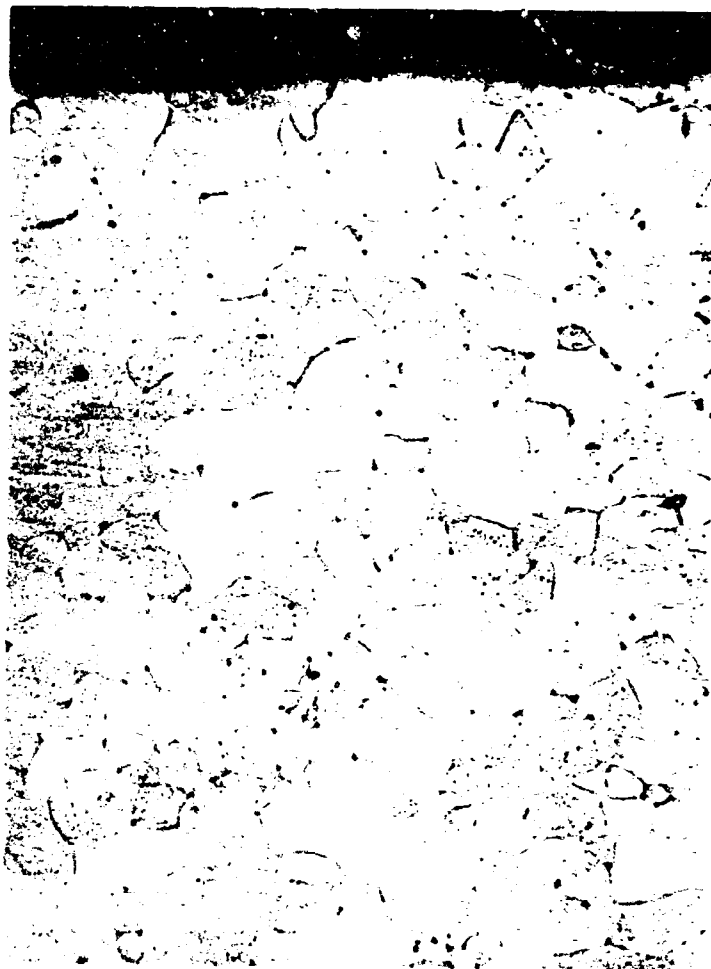


FIGURE 55. 2 PREHEATER, 200X, EXPOSURE  
TEMPERATURE,  $\sim 1000^{\circ}\text{F}$



FIGURE 56. 3 BOILER, 200X, EXPOSURE  
TEMPERATURE, 1350°F

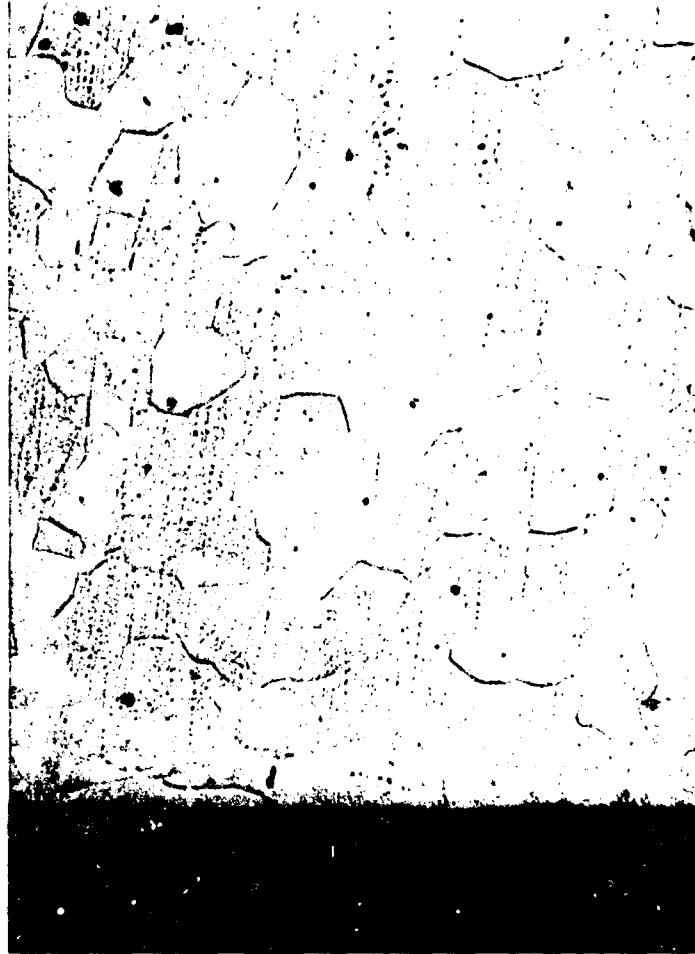


FIGURE 57. 4 BOILER, 200X, EXPOSURE  
TEMPERATURE, 1900°F



FIGURE 58. 5 SUPERHEATER, 200X, EXPOSURE  
TEMPERATURE, 1930°F



FIGURE 59. 6 SUPERHEATER, 200X, EXPOSURE  
TEMPERATURE, 1930°F

LONGITUDINAL SECTION (WITH WELDMENT)



TRANSVERSE SECTION



FIGURE 60. 7 SUPERHEATER OUTLET, 50X  
EXPOSURE TEMPERATURE, 1930°F



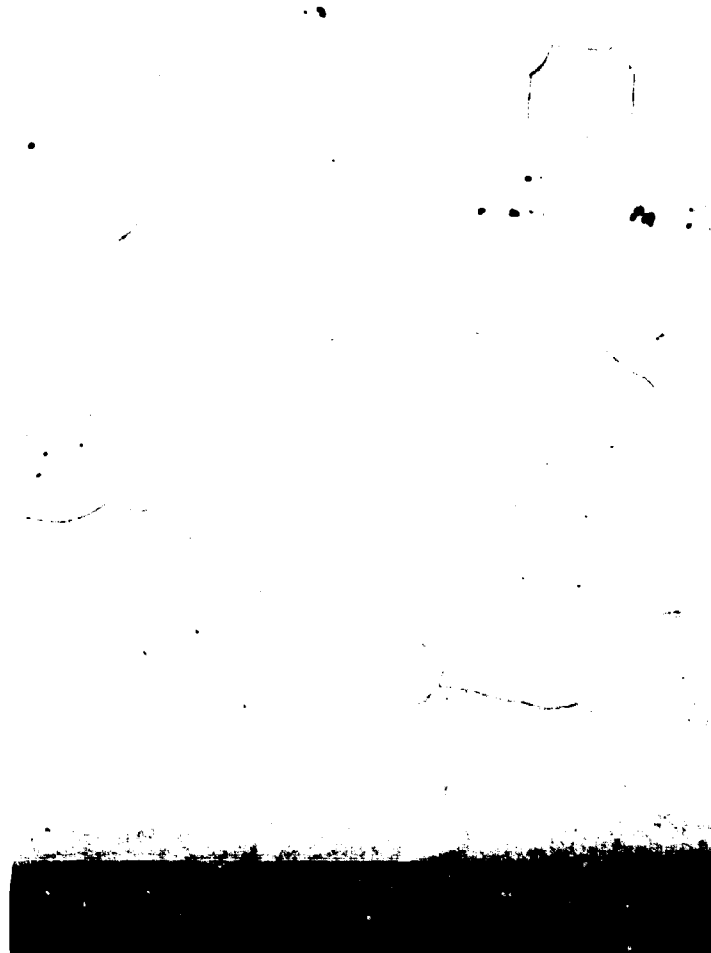


FIGURE 61. 8 CONDENSER OUTLET, 200X  
EXPOSURE TEMPERATURE, 212°F

APPENDIX II  
TWO-PHASE Cb + 1.0 w/o Zr LOOP  
PANEL DESCRIPTION  
AND  
OPERATING INSTRUCTIONS

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## CONTROL PANEL DESCRIPTION

### 1. MASTER

Turns on the control-panel power; must be on to operate anything on this panel.

### 2. START HEAT ON

These are the master heat switches.

### 3. START HEAT OFF

These control power to the main heaters only on start. Once the EM pump is started and flow has commenced, these switches become inoperative (indicated by the light going out). This allows the initial system heatup without power to the EM pump as it bypasses the pump interlock. Once flow is established, the "HEATER CUTOFF" switch (No. 4) must be used to cut power. The "START HEAT ON" circuit bypasses all Simplitrol circuits on the meter control panel and the low flow limiter (No. 22) on this panel. The heat-up can be started without sounding any alarms. The "HEAT ON" light (No. 20) will light when start heat is energized.

### 4. HEATER CUTOFF

A manual on-off switch, which controls the main loop heaters after flow is established, and the "START HEAT" switches are inoperative. This does not control the immersion heater. This switch lights automatically when it takes control. Push to cut off heat.

### 5. IMMERSION HEAT

This switch controls the 6-kw argon heater in the argon line at the recirculating blower discharge. The blower must be on (No. 9) before the immersion heater circuit can be energized. The "START HEAT ON" circuit must also be energized before the heater can be turned on; therefore, when the flow of potassium is established and the "START HEAT" circuit drops out, the immersion heater circuit will automatically be turned off. This heater heats the argon to preheat the loop for fill. Once potassium flow is established, this same circuit (HEATER OFF) is used for condenser cooling. The heater will also drop out when the blower is turned off during the heat-up cycle. The heater has no voltage control and will be on full bore during use. If any overheating of argon is noted, this immersion heat must be manually cut off.

## 6. LEVEL INDICATOR

This switch controls the power to the liquid-level indicator which will be described separately.

## 7. PUMP START "A"

This circuit cannot be energized until the "MASTER," "BLOWER," and "START HEAT" circuits are on. It supplies power to the EM pump by bypassing the low-flow limiter, which would normally prevent pump operation when flow drops off. When energized, it drops out the immersion heater and START HEAT circuit. Power to heaters is controlled with the heat cutoff switch (No. 4). With the "PUMP ON" switch energized, light No. 21 will light.

## 8. PUMP START "B"

After the flow is stable, this switch can be energized and the low-flow limiter can be put into operation. "PUMP START A" switch will automatically drop out at this time. It also turns on a timer motor, which sweeps the alarm control panel for trouble and sounds alarms, etc., when detected. This is now in full operating condition; heat and flow are now interlocked, and protection circuits are energized.

## 9. BLOWER

This switch must be on and the blower running before the immersion heater can be turned on, which makes it the second in sequence (the MASTER is first). This controls the chamber argon recirculating and cooling blower. To use this switch and start the blower, the Varidrive must be running. Speed is controlled by the "BLOWER SPEED INCREASE-DECREASE" switch (No. 11). When the blower is running, the blower indicator light (No. 12), located directly above the Varidrive switch (No. 10) will be glowing. See Varidrive controls No. 10, No. 11, and No. 12 for power to the blower motor.

## VARIDRIVE CONTROLS

### 10. VARIDRIVE

This is a switch to start or stop the Reliance Varidrive located outside the N.E. corner of the test cell that supplies a-c power for the chamber recirculating blower motor. It must be on before the blower runs.

### 11. BLOWER SPEED INCREASE-DECREASE

This toggle switch actually controls the speed of the Varidrive alternator that determines the blower speed. The Tachometer (No. 27) directly above this switch reads alternator speed and allows accurate resetting of the flow points.

## 12. BLOWER

This is an indication light only, and indicates that the blower motor is running [see "BLOWER" switch (No. 9)].

## 13. HEAT SELECTOR

This selector switch selects the main loop heaters--two in the preheat section, three in the boil-heat section, and three in the superheat section. After selecting the section and individual heater desired, heaters can be varied by using the "HEAT INCREASE-DECREASE" switch (No. 14). This selector switch also selects voltage and amperage readings for each heater, read on meters 23 and 24, as well as the temperature of boil thermocouple No. 29 and superheat thermocouple No. 35 sections only, read on temperature readout.

## 14. HEAT-INCREASE-DECREASE

This toggle switch is used to drive the motorized Variacs controlling the power to the main loop heaters, which increases or decreases temperature. The individual heater or section of heaters is selected by the heater selection switch and toggle switches (No. 15, No. 16, and No. 17). The "START HEAT ON" switch must be energized to get power to this circuit. Once the flow of potassium is established, the "START HEAT" circuit will drop out automatically and limiting Simplitrols will be in the circuits.

## 15. PREHEAT PARALLEL-INDIVIDUAL

This toggle switch allows selection of the changing temperature of the two main heaters in this section simultaneously by putting it in the "PARALLEL" position or individually by putting it in the "INDIVIDUAL" position and using "HEAT INCREASE-DECREASE" (No. 14). "HEATER SELECTION" (No. 13) must be in the corresponding section, but no particular heater number is necessary.

## 16. BOIL HEAT PARALLEL INDIVIDUAL

This toggle switch gives a selection of changing temperatures of the three main heaters in this section simultaneously by being put in the "PARALLEL" position or individually by being put in the "INDIVIDUAL" position, and using "HEAT INCREASE-DECREASE" (No. 14). The heater selector (No. 13) must be in the corresponding section but is not restricted to any number.

## 17. SUPERHEAT PARALLEL-INDIVIDUAL

This toggle switch gives selection of changing temperatures of the three main heaters in this section simultaneously by putting it in the "PARALLEL" position or individually by putting it in the

"INDIVIDUAL" position, and using "HEAT INCREASE-DECREASE" (No 14). The heater selector (No. 13) must be in the corresponding section but is not restricted to any number.

#### 18. PUMP INCREASE-DECREASE

This switch increases or decreases voltage to the EM pump and thereby increases or decreases the flow of potassium through the loop. "PUMP START" "A" (No. 7) or "B" (No. 8) must be energized before power is available.

#### 19. VOLTMETER, 0-150, 0-300

This toggle switch selects one of the two voltmeters on which the pump voltage can be read--(No. 25) 0 to 300-volt range. or (No. 26) 0 to 150-volt range.

#### 20. HEAT ON

An indication light only, indicating when power is on to the main loop heaters. This lights when the "START HEAT ON" (No 2) switch is energized.

#### 21. PUMP ON

An indication light only, indicating when power is on the EM pump. It lights when "PUMP START" "A" or "B" is energized

#### 22. FLOW

A microammeter with a low set point that can be adjusted will read the signal from the flowmeter and is intended to shut down all power when the flow drops below the point selected by the limiter needle. Flow is not to be read on this instrument, but from the Hewlett-Packard VTVM located below the alarm Simplitrol panel, as it has much greater accuracy. Scale on the VTVM can be adjusted between 1(001) and 3(003) millivolts as needed. This meter will be used as an indication only and as the low flow limiter. Do not set this flow limiter until the "START HEAT ON" switch is de-energized and limit switches are in the system.

#### 23. HEAT, AMPERES

An a-c ammeter to read amperes of heaters as selected by the "HEATER SELECTOR SWITCH" will be used.

#### 24. HEAT, VOLTS

An a-c voltmeter to read the voltage of heaters as selected by the "HEATER SELECTOR SWITCH" will be utilized.

## 25. and 26. PUMP, VOLTS

A-c voltmeters of 0- to 300- and 0- to 150-volt ranges will be used to read EM pump voltage as selected with toggle switch No. 19.

## 27. RELIANCE TACHOMETER

This tachometer reads rpm of the alternator on the Reliance Varidrive, which furnishes power to the recirculating blower motor. It does not read rpm of the blower. It will enable flow points to be reset or repeated if the rpm of the alternator is noted previously.

## 28. LIQUID LEVEL-PROBE SELECTOR

This selector chooses the individual probes in the surge tank. The probe readings are indicated on Ballentine Voltmeter No. A-19. To reach each position individually, turn the pointer (No. 28) to the desired position and place the scan switch (No. 30) in the "MANUAL" position. Then push switch No. 29 to get a reading. One indicator light (No. 31, 37, 33, 34, and 35) will light corresponding to the probe being read. To read another point, turn the selector to another position and press the No. 29 switch again.

## 29. PUSH TO READ

This switch must be pressed to obtain a reading on a manual selection and must be pressed for each desired reading.

## 30. SCAN SELECTOR

Place this switch in the manual or automatic position as desired. The switch must be in "MANUAL" position for individual readings. Place the switch into "AUTO" position for automatic scanning. On the "AUTO" position, no other switches need be energized.

## 31. - 35. INDICATOR LIGHTS

These light when that pickup is showing on the voltmeter.

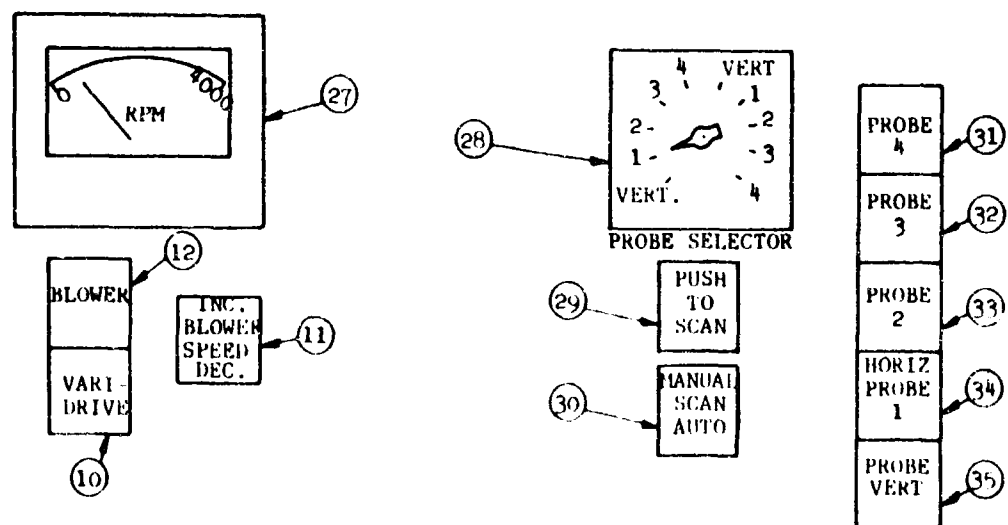
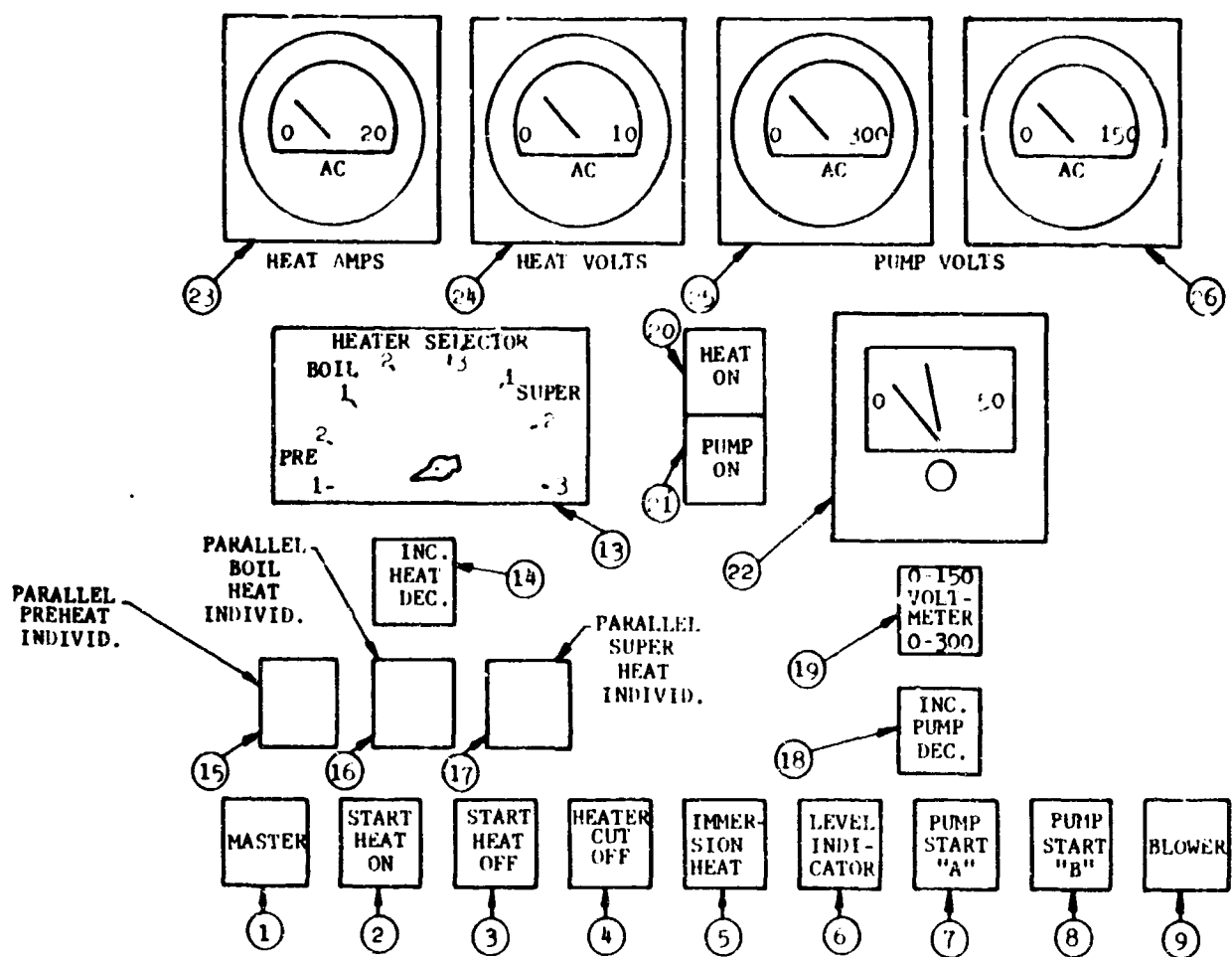


FIGURE 62. CONTROL PANEL



## ALARM CONTROL PANELS

There are lights behind every switch. When a section (usually half) of a switch is lighted, an alarm also sounds, indicating trouble. These areas will be described separately. When a light goes on and the alarm sounds, the alarm can be and should be silenced at once by pressing the switch that is lighted. The light will remain on until the fault is corrected, but you have released the alarm so that it may be available for other problems. Two conditions may prevail. They are:

### 1. Alarm Condition

An intermittent sounding of the alarm, and an indicator light on the panel. When located, the alarm is silenced by pressing the lighted switch on the panel. The alarm will turn off automatically if the fault is corrected; but by the alarm being turned off manually, it is available for other conditions.

### 2. Shutdown Condition

A continuous sounding of the alarm means that all control power and therefore all loop power has been turned off automatically. Panel power will stay on but will indicate a problem. The fault must be found, corrected, and restart made on the control console, if possible. Only one condition will cause shutdown--loss of the flow of potassium, triggered by flowmeter low-flow limiter No. 22 on the control console.

## SWITCH AND INDICATOR LIGHT - INDICATION

### 1. Preheat Section

- |    |                      |  |
|----|----------------------|--|
| A. | Low & No. 25 Control | The preheat section temperature is below the low-limit set point on the corresponding Simplitrol, meter control panel (No. 7). Light and intermittent alarm condition. |
| B. | Power                | Loss of power to control circuit or Simplitrol of preheat section. Light and intermittent alarm condition.   |

### 2. Boil Section

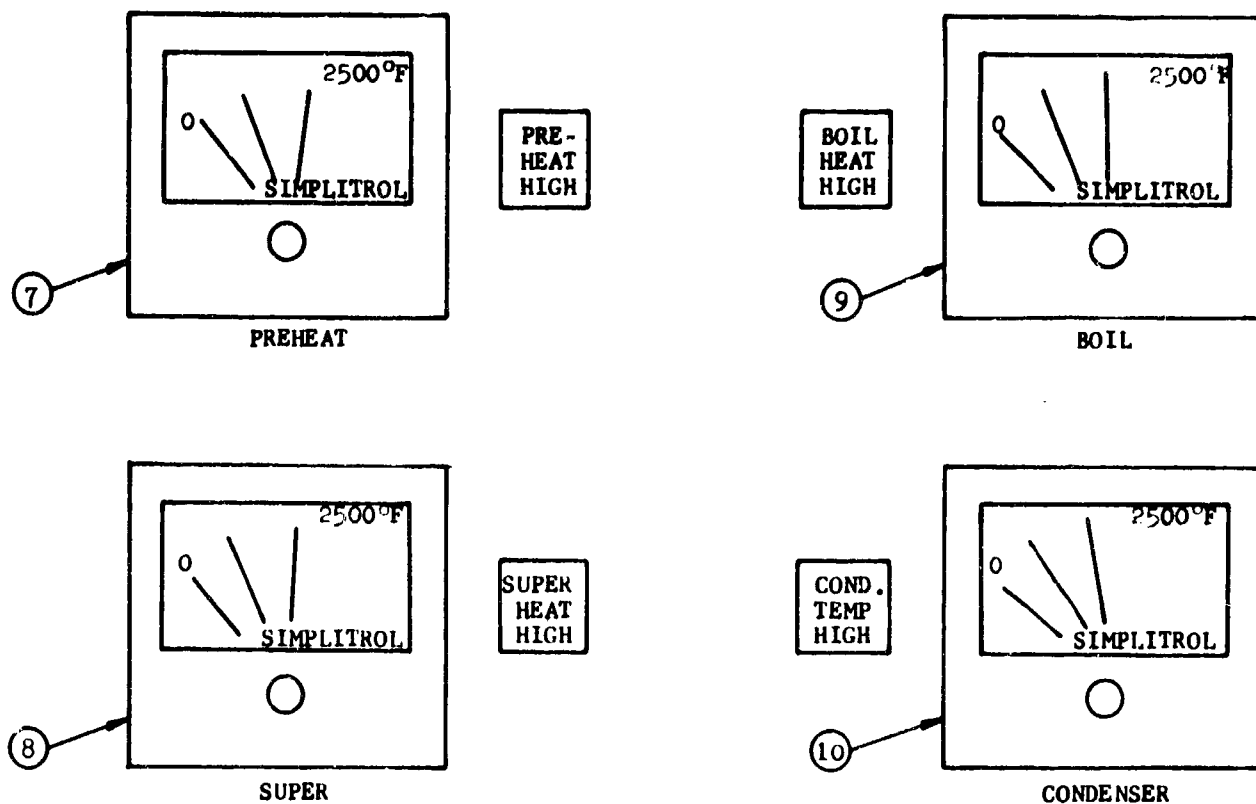
- |    |     |   |
|----|-----|---|
| A. | Low | Boil section temperature is below the low-limit set point on the Simplitrol (No. 9). meter control panel. Light and intermittent alarm condition. |
|----|-----|---|

- B. Power Loss of power to control circuit or Simplitrol of boil section. Light and intermittent alarm condition.
3. Superheat Section
- A. Low Superheat section temperature is below the low-limit set point on the superheat Simplitrol (No. 8). Light and intermittent alarm condition.
- B. Power Loss of power to the control circuit or Simplitrol if superheat section. Light and intermittent alarm condition
4. Condenser
- A. High & No. 5 Control Indicates that condenser outlet temperature is too high. Light and intermittent alarm condition. Indicates loss of power to the condenser Simplitrol circuit. Light and intermittent alarm condition.
5. A. Blower Indicates low pressure on the argon regulating blower; light and intermittent alarm condition, indication only-- nothing is turned off.
- B. Flow Power Indicates loss of power to flowmeter low-flow limiter. Light and intermittent alarm condition.
6. A. Low Flow Indicates low or no flow of potassium below the set point on flowmeter low flow limiter (No. 22 Control Console). This is a shutdown condition--steady alarm, loss of all power, reset on control panel, check loop temperature for possible freeze-up.
- B. Condenser Low Temperature Indicates that the condenser discharge temperature has reached the low-limit set point and is ready to freeze the potassium. This is a steady alarm condition and warns of the possibility of freeze-up and losing flow (shutdown condition). Low temperatures must be raised to correct the situation

## METER CONTROL PANEL

<u>Simplitrol</u>	<u>Set Points</u>	<u>Condition</u>
7. Preheat Control No. 25	Hi-Limit	Lights preheat high indicator, cuts off heaters, and resets them. Intermittent alarm condition.
	Lo-Limit	Lights alarm light (No. 1); sounds intermittent alarm.
8. Boil Heat Control No. 33	Hi-Limit	Lights boil heat high indicator. Cuts off heaters and resets them. Intermittent alarm condition.
	Lo-Limit	Lights alarm light (No. 2); sounds intermittent alarm.
9. Superheat Control No. 37	Hi-Limit	Lights superheat high indicator. Cuts off heaters and resets them. Intermittent alarm condition.
	Lo-Limit	Lights alarm light (No. 3); sounds intermittent alarm.
10. Condenser Temperature Control No. 5	Hi-Limit	Lights condenser high-temperature indicator and (No. 4) intermittent alarm condition.
	Lo-Limit	Lights alarm light (No. 6B), sounds continuous alarm condition. Does not shut down the loop.

**NOTE:** The hi-limit set points on the above Simplitrols sound the intermittent alarm and light the high-temperature lights beside the Simplitrols. They also shut off the corresponding heaters on a time-delay relay which, after a time lapse, turns the heaters back on again and cycles them on and off. Corrections should be made on the appropriate heater controls to decrease the temperature.



METER CONTROL  
P47-78A-236

HEWLETT-PACKARD VTVM FOR  
READING FLOWMETER SIGNAL

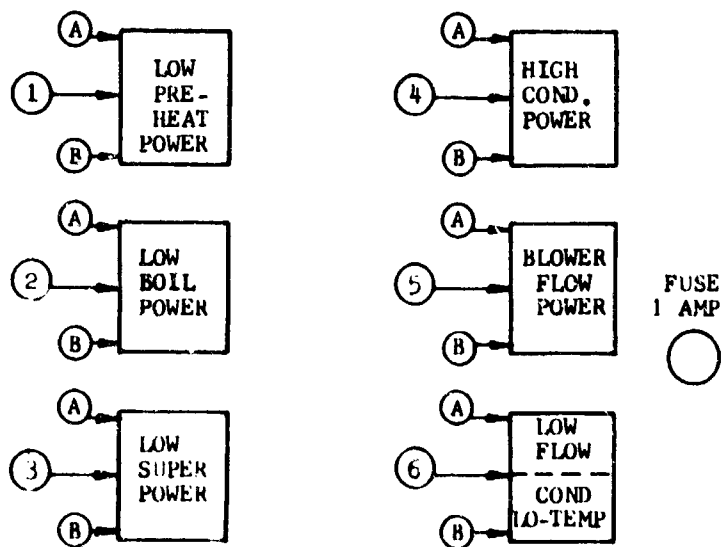


FIGURE 63. ALARM CONTROL PANEL

## DIGITAL DATA SYSTEM

The procedure for operation of the digital system and the correct settings for the two-phase loop are as follows, starting with the top instruments and proceeding downward to the last instrument in the console.

Section 1	Counter that indicates the scanner switch number and position. No adjustments.		
Section 2	Digital Voltmeter	Range setting	0.1 volt
		Function	Volts
		Sensitivity	Clockwise
		Sample period	0.1 sec.
		Sample rate	Stop
		Power switch	On
Section 3	Alarm System - Disable button overrides alarm sequence and gives one set of readings when pushed at alarm condition, then resumes the normal cycle.		
Section 4	Digital Clock	Time base	60
		Off-set-operate	Operate (after correct time is set)
		Print control	Clock
		Print rate	As desired
Section 5	Recorder	Power	On
		Record	On
		Space selection	1
Section 6	Master Control	Power	On
		Start	Manual operation of the scanner-- may be used any time to reset and start the scanner.
		Reset	

Section 6    Master Control  
              (Contd.)

Alarm light

Alarm condition  
on loop when  
lighted--  
overrides timing  
sequence and  
records auto-  
matically twice,  
then resets to  
normal scan.  
Light stays on  
until the fault  
is corrected.

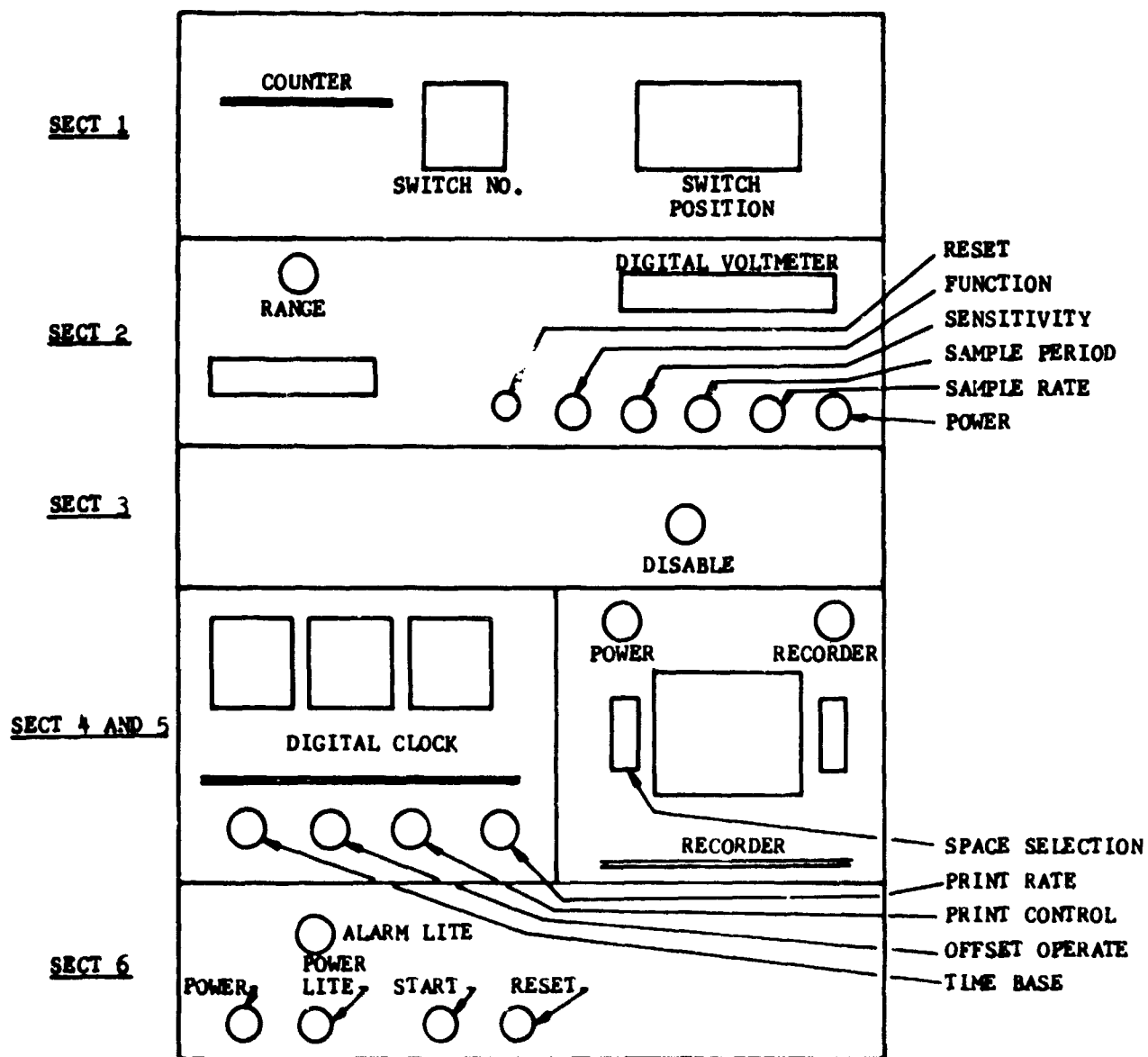


FIGURE 64. DIGITAL DATA CONTROL CONSOLE

## LIQUID-LEVEL GAUGE OPERATION

The horizontal probes all have about the same output in millivolts, with the vertical probe output being considerably higher. When any probe is submerged in potassium its output is reduced, so it is simple to determine the interval in which the level occurs. Since Probe No. 4 is on the bottom, the level between No. 2 and No. 3 would produce readings such as: No. 4 and No. 3,  $1/2$  mv; No. 2 and No. 1, 2 mv. These are not the readings that will result, but only examples.

The horizontal probes indicate the interval in which the level occurs and are used to calibrate the vertical probe, which gives a continuous output that is proportional to level. Whenever the level is changing, such as when going from one to two phase, watch the horizontal probes; and when one changes and stabilizes, the level will be at that point. Switch over to the vertical probe and record the output. Note that the probe is  $1/4$  inch in diameter, so plot the points for the level either barely touching or barely covering the probe. This should be apparent from which way the level is moving. Plot the level versus vertical probe output on a calibration chart similar to the attached, which is an example only. The vertical probe output can be determined by actual operation only. Plot in a best-fit line or curve. Refer to this calibration line to determine the level at any time. (Figure 25).

This calibration may change with time, so obtain new calibration points whenever possible.



## VACUUM PULLDOWN AND OUTGASSING

It cannot be accurately determined how much time will be needed to pull down the chamber and system components and outgas sufficiently. Determination will have to be made by Engineering during the cycle. Previous work completed: Complete installation and triple check of all systems and components as well as electrical system and instrumentation. Chamber is closed and ready to pull down.

1. Start procedure with all valves closed. Turn the KD-30 roughing pump at switch "RP" on the east wall in the test bay (see diagram).
2. Open Valves S A, D, R, and Q, 3 and 4 and evacuate the molecular sieves to 100 microns. Crack R<sub>1</sub> and R<sub>3</sub>, then bleed through Valve 1 for 1 minute. Close Valve 1.
3. Open Valve B and start the KC-5 roughing pump (the switch is located on the pump).
4. Turn on cooling H<sub>2</sub>O manually (open 32 and 33). Check H<sub>2</sub>O flow at the discharge outside northeast corner of the test cell. Turn on PMC-721 diffusion pump power (the switch is on the east wall panel).
5. Close Valve A and open Valve C. This puts the diffusion pump system on line and cuts the KD-30 roughing pump out of the system. Keep the KD-30 pump running.
6. Evacuate sieves down to 1 micron; hold for 1 hour.
7. Close Valve C, and keep the diffusion pump system running.
8. Check the decay of the molecular sieve system. Repeat the pump-out cycle, if necessary, at the discretion of the engineer in chart (E.I.C.).
9. Close Valves R and Q.
10. With the argon lines assumed to have been previously bled or evacuated (and if not certain, perform this first), pressurize Regulator R<sub>1</sub> to 200 psig and Regulator R<sub>3</sub> at 5 psig; open Valve 1, and fill the molecular sieves with argon to 5 psig maximum. Close Valves 3, 4, and 1.

11. Open Valves 12, 13, 5, 6, M, N, and A. Evacuate purifiers to 100 microns. (Crack Valve 2 and bleed argon for 1 minute, then close Valve 2.) Close Valve A and open C and continue pumpdown to 1 micron. It may be necessary to take one set of purifiers at a time--Valve 12 first, and then Valve 13, and pull down the manifold up to Valves L and K.
12. Close Valves 12 and 13 and fill the system with argon (by opening Valve 2) to 5 psig. Close Valves 5, 6, N, and M. Then close Valve 2. Close Valve C.
13. Open Valves E, F, G, H, A, and 19 and evacuate the chamber and blower box by using a standard\* roughing and diffusion cycle. At this time connect a mass spectrometer at Valve 22 and carefully check the chamber and all components for any helium leakage. When satisfactory, place the radiant heaters around the chamber and Close Valves 22, C, D, and E. Be careful in doing this in order that no transformers or other equipment will be damaged by the heat. Open Valves 21 and A. Open K12, K11, K9, K8, K7, and K6. Pressurize R to 1 psig and crack K14. Close K14. Close Valve A and open Valve C when the vacuum reaches 100 microns. Read pressure at K13 when in range. Close Valve C (when 1 micron is reached). Close Valve 21 and pressurize to 1 psig through K14. Then close K14.
- 13-A. Repeat evacuation and purge as follows: open Valves 21 and A. When 100 microns are reached, close Valve A and open Valve C. When 1 micron is reached, close Valve C and pressurize to 1 psig by opening K14. Then close K14.
- 13-B. Repeat 13-A. Then close K14 and open Valves 21 and A. Close Valve A and open Valve C when the pressure reaches 100 microns and pull to 1 micron; then close Valves 21 and C.
14. Turn on the loop heater selector switch (No. 13) and switches No. 15, 16, and 17 in "individual" positions; start power to each heater with "HEAT INCREASE-DECREASE" switch No. 14. Log data as to amps and volts and to each heater at every power change. Hold the maximum temperature to 300°F by monitoring manual readout. (Step 15 should start when the loop heaters are turned on.)

\*Standard Vacuum Cycle: Pull down to 100 microns with KD 30 roughing pump through Valve A. Close A and open Valve C at 100 microns and pull down to 1 micron via the diffusion pump and KC-5 pump.

15. Turn on the radiant heaters and log pressure readings at least every 30 minutes. (Check the thermocouple on the chamber wall to hold bakeout at 300°F maximum) (T/C 112, 116 and 90, 69, and 68). Continue bakeout at the discretion of the E.I.C.
16. Close Valves E, G, and 19 and open Valve 8 to fill the chamber to 1 psig pressure with argon by regulating R<sub>3</sub> and Valve 2. Close Valve 2 when pressure is reached.
17. Close Valve 8. Open Valves A and E to evacuate the chamber again, with the radiant heaters still on.
18. Repeat steps 16 and 17 once more (for a total of three pulldowns). The last pulldown should be to 1 micron, by using a diffusion pump. Close Valves D and C; open Valves 21 and A and evacuate the loop to 1 micron, with the standard vacuum cycle used.\* At this time repeat step 16, with the argon pressure raised to 5 psig. Open Valves G and 19.
19. At this time the chamber and purification system have been evacuated, outgassed, and pressurized. The loop is still under vacuum and pumping; interrupt loop pumping by closing Valves C, 19, and K12. Open Valves 28, 24, and A and evacuate the line down to 2,000 microns (read at Valve 22). Crack Valve 23 to purge the line with argon for 1 minute. Close 24 and 28 and open 23 and 19.
20. Pump on manifold to K12 until 100 microns is reached at K13; open K12 and check the pressure, then close Valve A and open Valve C. Turn off the radiant heaters. Leave the loop heaters on at 300°F with the vacuum system pumping on the loop. The chamber is now under argon pressure and ready for purification system operation.

#### VALVE DESIGNATIONS

- A - 2" CVC-VST, between cold traps, inlet to KD-30
- B - 2" CVC Ball, diffusion pump discharge
- C - 4" CVC-VST, diffusion pump inlet
- D - 3" CVC-VST, upstream of vacuum pumps and cold traps

\*Standard Vacuum Cycle: Pull down to 100 microns with KD-30 roughing pump through Valve A. Close A and open Valve C at 100 microns and pull down to 1 micron via the diffusion pump and KC-5 pump.

- E - 3" CVC-VST, inlet to blower box and large chamber
- F - 3" CVC-VST, blower discharge into chamber, EM pump
- G - 3" Hi-Flow Needle, Hi-Temperature argon from purifiers into chamber
- H - 2" NRC Bellows, blower discharge into chamber, flowmeter
- I - 3" CVC-VST, argon inlet to right-hand molecular sieve
- J - 3" CVC-VST, argon inlet to left-hand molecular sieve
- K - 3" CVC-VST, discharge from right-hand molecular sieve
- L - 3" CVC-VST, discharge from left-hand molecular sieve
- M - 3" CVC-VST, inlet into low-temperature purifiers
- N - 3" CVC-VST, inlet into high-temperature purifiers
- O - 3" Hi-Flow Needle, discharge from low-temperature purifiers
- P - 3" Hi-Flow Needle, discharge from high-temperature purifiers
- Q - 2" NRC Bellows, vacuum inlet to right-hand molecular sieve
- R - 2" NRC Bellows, vacuum inlet to left-hand molecular sieve
- S - 2" CVC-VST, KD-30 inlet
- T - 6" NRC, Argon Chamber Discharge
- U - 6" NRC, Recirculating Blower Discharge
- V - 3" CVC-VST, Vacuum Valve on Chamber

- 1 - 1/4" 482 Bellows, argon supply to molecular sieves
- 2 - 1/4" 482 Bellows, argon supply to purifiers
- 3 - 1/4" 482 Bellows, argon supply to right-hand molecular sieve
- 4 - 1/4" 482 Bellows, argon supply to left-hand molecular sieve
- 5 - 1/4" 482 Bellows, argon supply to low-temperature purifiers
- 6 - 1/4" 482 Bellows, argon supply to high-temperature purifiers
- 7 - 1/4" 482 Bellows, argon supply into 3" discharge from purifiers
- 8 - 1/4" 482 Bellows, argon supply into 3" line at blower box
- 9 - 1/4" 482 Bellows, roughing pump cold trap drain
- 10 - 1/4" 482 Bellows, Pirani vacuum-gauge port between cold traps
- 11 - 1/4" 482 Bellows, diffusion pump cold-trap drain

- 12 - 1/2" Superior, vacuum inlet at top of low temperature purifiers
- 13 - 1/2" Superior, vacuum inlet at top of high-temperature purifiers
- 14 - 1/2" Superior, inlet to gas analyzer
- 15 - 1/2" Hi-flow Bellows, gas sampler, heater at flowmeter
- 16 - 1/2" Hi-flow Bellows, gas sampler, heaters at bottom corner
- 17 - 1/2" Hi-flow Bellows, gas sampler, heaters at top corner
- 18 - 1/2" Hi-flow Bellows, gas sampler, discharge at heaters
- 19 - 1/2" Hi-flow Bellows, gas sampler, 3" discharge from purifiers
- 20 - 1/2" Superior, vacuum tap downstream of KD-30 cold trap
- 21 - 1/2" Superior, vacuum tap, diffusion pump cold trap
- 22 - 1/4" 482 Bellows, vacuum tap, upstream of diffusion pump cold trap
- 23 - 1/2" Hoke TY 477, shutoff to Hg pressure
- 24 - 1/4" Hoke 482, evacuation valve for pressure-relief line
- 25 - Inlet to residual gas analyzer
- 26 - Inlet to moisture monitor
- 27 - 1/2" Superior, Pirani Outlet, back of chamber
- 28 - 1/4" Hoke 482, Vacuum source to sampling lines and Hg pressure relief, RGA, H<sub>2</sub>O Mon., etc.
- 29 - Grease gun outlet
- 30 - H<sub>2</sub>O - Argon Hx., H<sub>2</sub>O inlet
- 31 - H<sub>2</sub>O - Argon Hx., H<sub>2</sub>O outlet
- 32 - Diffusion pump H<sub>2</sub>O inlet (cooling)
- 33 - Diffusion pump H<sub>2</sub>O outlet (cooling)
- 34 - Sampling System cutoff valve
- 35 - Upstream cutoff valve, argon
- 36 - Downstream cutoff valve, argon
- 37 - 1/4" Hoke Bellows, Manual Inbleed for KS-13
- 38 - 1/4" Hoke Bellows, Argon Inlet to Blower Box
- 39 - 1/4" Hoke Bellows, Argon Inlet to Chamber

## PURIFICATION SYSTEM OPERATION

The purpose of the purification system is to obtain the purity of the argon atmosphere needed to prevent oxidation of the Nb/Zr at high temperatures. There are two sets of purification heaters as shown on SK-LT31. Each set has a 1500°F and an 800°F furnace. The sections of the tanks above the heaters are filled with titanium chips to absorb oxygen and nitrogen. There are also two molecular sieves, which the argon is passed through, to extract any moisture and hydrocarbon atmosphere. There are two complete sets of purifiers and sieves - one is for use; the other as a spare system if the first needs regeneration. The purifier blower merely circulates the atmosphere through the purification system.

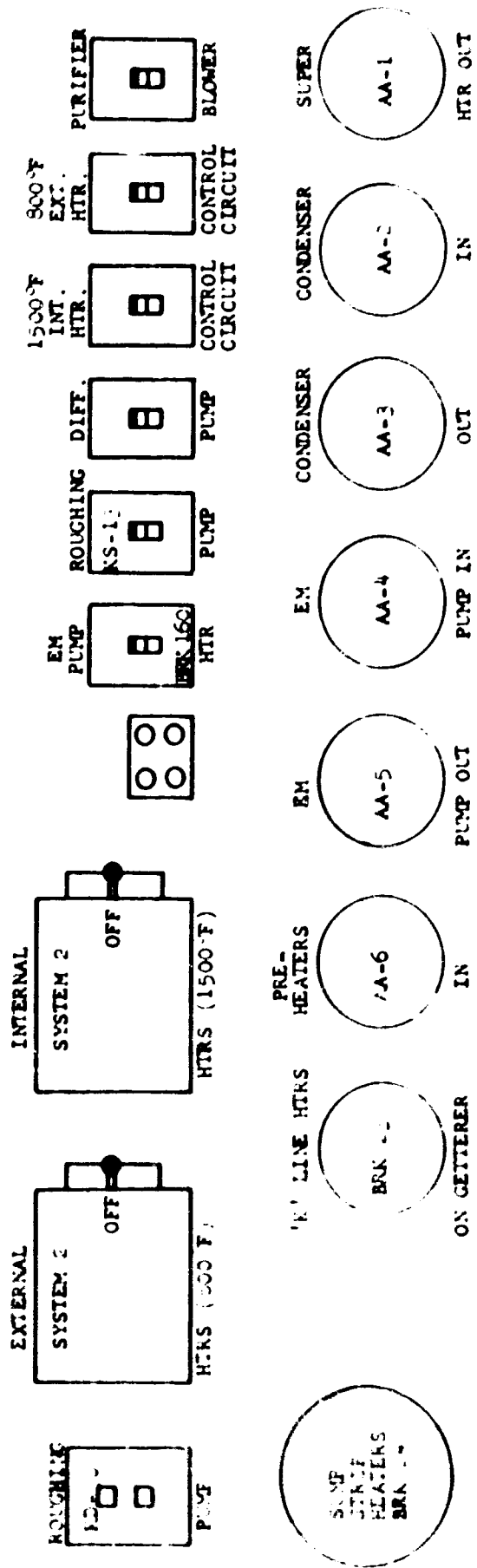
### OBTAINING ATMOSPHERE AND OPERATION OF SYSTEM

1. The chamber and purification system have been outgassed and are now at 5 psig of argon pressure, ready for purification. All purification valves are closed.
2. Open Valves I, K, N, P, and G. This opens a path for argon to flow through one molecular sieve and one set of purifiers (west set of purifiers, east sieve).
3. Start the purifier blower, 'PB' switch on the east wall panel (see sketch of panel).
4. Open Valves 15, 16, 17, 18, and 19. Throttle back on G to distribute the flow. Flow argon for 15 minutes, then close Valves 15, 16, 17, 18, and 19 and open G wide.
5. Turn on the furnace heaters for the purifiers argon is flowing through; Pur-1, in this instance. Check circuit breakers No. 193, outside north wall, making sure 1-1 and 1-2 Variacs are at 0 power. Then turn on Pur-1 switch on the east wall panel, which energizes the thermostat control circuit for this set of purifiers.
6. The Variacs can then be started up in power to raise the purifier furnace temperatures. The temperatures will be controlled automatically when limits are reached by the heater thermostat control (built in). Bring the argon purification heaters up at a rate of 600° per hour.

7. The chamber recirculating blower will also be run to circulate argon through the blower box, ducting, and argon-H<sub>2</sub>O heat exchanger. It is controlled from the console in the Control Room (Control Panel SK-LT32). To start the blower, energize the master switch (No. 1), turn on Varidrive, switch (No. 10), run rpm on tachometer (No. 27) down near 0, with blower speed switch No. 11 (the blower is not yet running). The tachometer actually reads rpm of the Varidrive alternator which supplies power to the blower motor. Energizing blower switch (No. 9) starts the blower once the Varidrive is running. Adjust blower speed as needed.
8. With power to the blower, the immersion heater can be turned on. Valves F and H may be opened for more flow, or better distribution of flow into the chamber from blower discharge. Be sure that the high-temperature limits listed for the EM pump and flowmeter are not exceeded (thermocouples 55, 56, and 57 on the EM pump and thermocouples 58 and 59 on the flowmeter). Maximum temperature is 350°F.
9. Argon is now circulating through the chamber and blower box and the purification system. After 1 hour, cut back on the purification heaters to half temperature (400° and 800°). Open Valves 15, 16, 17, 18, and 19. Throttle Valve G to distribute flow. Flow for 15 minutes. Instrumentation personnel will start the sampling system and will monitor moisture, oxygen, and other gases in the argon atmosphere (through Valves 14, 25, and 26). When the argon is at an acceptable level established by engineering, the next step will be weld annealing. At this time the potassium purification cycle in the sump should be started (16 hrs at 1100°F). Argon pressure may be increased, if not sufficient, by flowing argon through regulator R<sub>3</sub> and Valves 1 and 3.

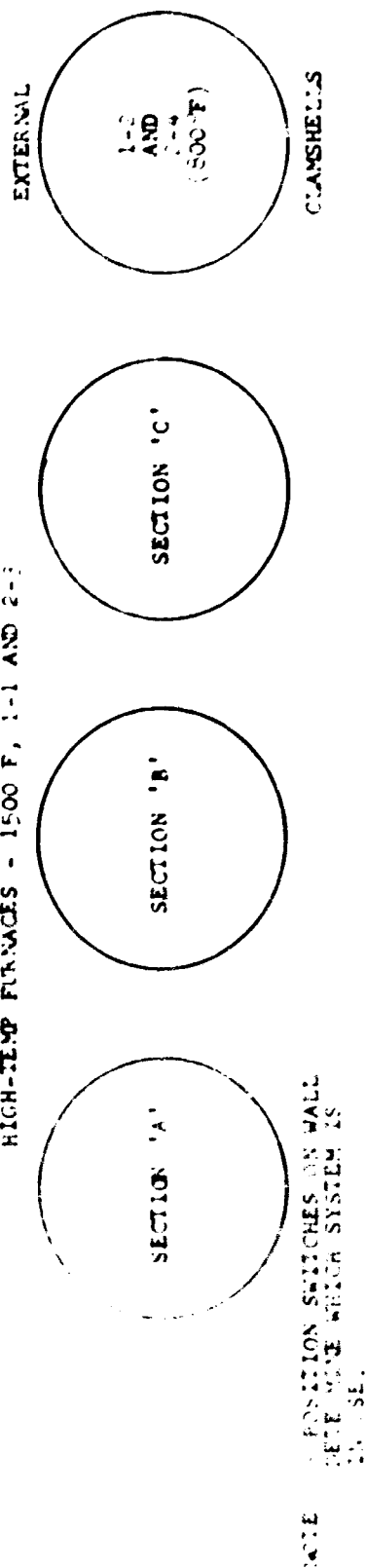
NOTE: Since the chamber recirculating blower is used in this instance for additional heat by passing argon over the immersion heater, it should not be necessary to flow water through the argon H<sub>2</sub>O heat exchanger located under the chamber. During test, this is to be used to keep the EM pump and flowmeter temperatures below the high limits set by Engineering (350°F).

# CONTROLS ON EAST WALL OF TEST BAY



## VARIACS ON EAST WALL

HIGH-TEMP FURNACES - 1500 F, 1-1 AND 2-2



NOTE: POSITION SWITCHES ON WALL DETERMINE WHICH SYSTEM IS IN USE.

FIGURE 65. VARIACS ON FLOOR



## GETTERING SUMP CHARGE

The high-purity potassium has been purchased in 100-lb shipments with Hoke 445 (K-1) Valve and Hoke 482 Argon Valve (K-2). Instrumentation will attempt to use a liquid-level device on the shipper. The sump tank, sampling legs, and fill manifold should have been previously leak-checked, connected to the system, and evacuated.

1. Crack shipper Valve K-2 and vent off excess cover gas. If the shipper is not under positive pressure, reject the shipper and use the spare shipper. Close K-2.
2. Connect the argon line to Valve K-2; leave the fitting loose for gas venting.
3. Pressurize the argon line slightly (1 psig) and allow this gas to leak through the fitting for 5 minutes. Then tighten the fitting and shut off the argon. Close K-2.
4. Connect the fill line from K-1 to K-3 using 316 S.S. and Swagelok fittings.
5. Open K-1, K-3, K-4, K-9, K-12, 21 A and S. Start the roughing pump and evacuate the line from the shipper, using a standard vacuum cycle.
6. Leak-check the fill system when 1 micron is reached.
7. Close K-4, maintaining the loop under vacuum pumping.
8. Do not backfill with argon because it could get into the loop.
9. Fill-lines are now connected, evacuated, leak-checked, and held under vacuum.
10. Close K-1.
11. Connect Thermo-Coax heaters and fiber-glass insulation around shipper and fill-lines. The thermocouple can be tacked on at this time also (after leak-check).
12. Turn on the heaters; heat the shipper and gettering sump to 300°F and hold for 4 hours.
13. Crack K-1 and allow potassium to flow into the gettering sump.

14. Fill the gettering sump until the spark probe indicates contact at the 4 lb mark. Then close F-1 and cut the heaters.
15. Open K-5 and K-14 and pressurize through R1 and R2 with argon to K-1 (1 psig). Open K-1 and allow argon to blow back, clearing the fill line. Close K-1, K-5, K-14, and R2.
16. Bring the gettering sump up to 1400 F after being at 600 F for 1 hour. Hold at 1400 F for 16 hours. Then reduce temperature to 400 F.
17. Potassium is now gettered and ready for fill of loop.

## WELD ANNEALING

Previous steps completed: Outgassing, Purification System Operation

Control panel switches on: Master, immersion heater, level indicator, blower purification system running.

1. The argon atmosphere has just been checked for purity and accepted. The purification system is in operation, and the chamber recirculating blower and immersion heater are both in operation.
2. The annealing heaters can be turned on and the cycle started. Make sure all six Variacs are on 0 power first. Go to the electrical cabinet outside (north side of new northeast corner) and turn on breakers No. 106, 107, 108, 109, 110, and 111. The Variacs can then be turned up slowly.
3. The rate of temperature increase is to be 500°F per hour. Welds are to be annealed for one hour at 2200°F. Heater power shall then be decreased and stabilized at 400°F. Everything is now ready for another check of the purity of the atmosphere before proceeding to the preheat cycle.

AA HEATER	IDENTIFICATION
AA-1 (Super Heat Out)	37-38 - Tube, Control 39-40 - Tube, Readout 41-42 - Adiabatic, Readout 43-44 - Adiabatic, Readout
AA-2 (Condenser In) (Test Chamber Out)	45-46 - Tube, Readout 47-48 - Tube, Readout 49-50 - Adiabatic, Readout 50-51 - Adiabatic, Readout
AA-3 (Condenser Out)	1 - 2 - Tube, Readout 3 - 4 - Adiabatic, Readout
AA-4 (EM Pump In)	5 - 6 - Tube, Condenser Out Control 7 - 8 - Tube, Readout 9 - 10 - Adiabatic, Readout 11-12 - Adiabatic, Readout
AA-5 (EM Pump Out)	13-14 - Tube, Readout 15-16 - Adiabatic, Readout
AA-6 (Preheater In)	17-18 - Tube, Readout 19-20 - Tube, Readout 21-22 - Adiabatic, Readout 23-24 - Adiabatic, Readout

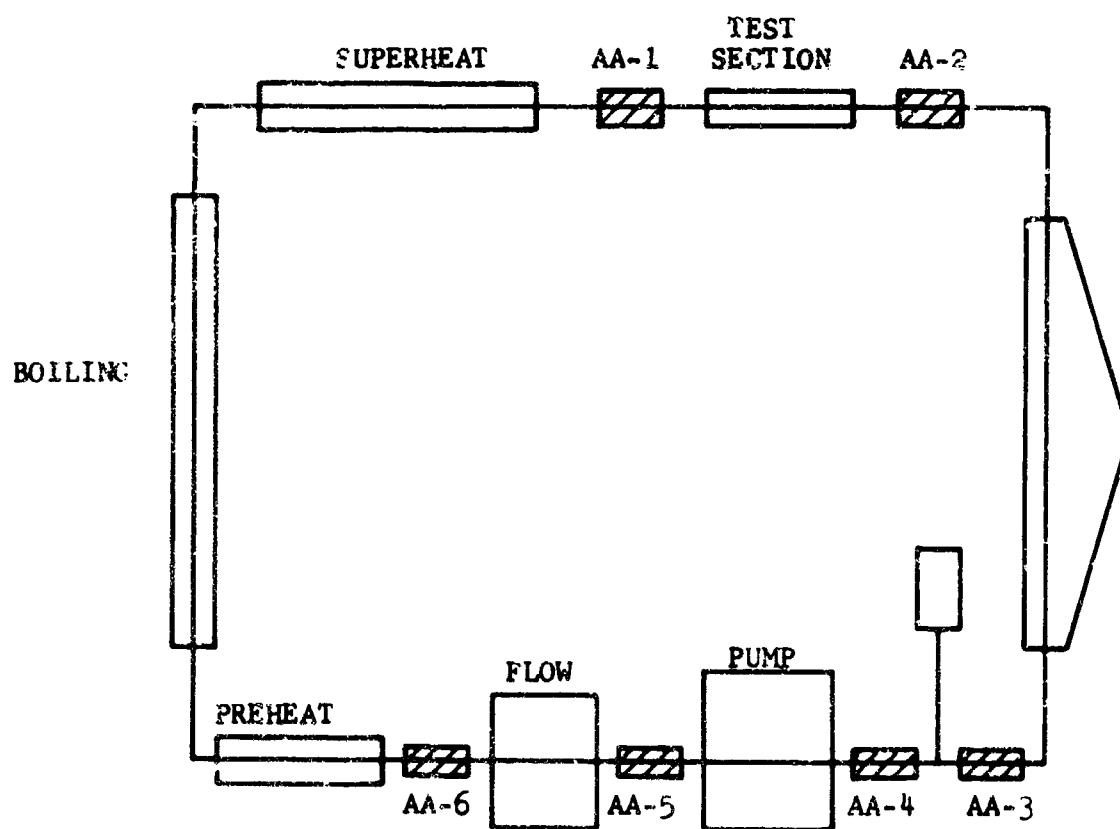


FIGURE 66

## PREHEAT CYCLE

Previous steps completed: Outgassing, purification system operation, and weld annealing.

Control panel switches on: Master, immersion heat, level indicator, blower. Purification system running, anneal and A. W. Heaters on.

1. The weld annealing procedure has just been completed. The power on the six annealing heaters has been reduced to stabilize the temperatures to approximately 400°F.
2. The main loop heaters are still on at 300°F.
3. With the heater selector switch (No. 13) and switches 15, 16, and 17 in "individual" positions, increase the power to each heater with "HEAT INCREASE-DECREASE" switch (No. 14). Log data as to amps and volts to each heater at every power change.
4. Stabilize the tube temperature at approximately 400°F.
5. Turn on Variacs to wrap the heaters that have been installed around transfer lines, valves, and Xmas tree. The sump has hot-soaked at 1400°F for 16 hours. Transfer lines, valves, and Xmas tree should be soaked at 250°F to 300°F.
6. The blower must be on with the immersion heater on. Flow of hot argon from the immersion heater should be directed through the duct around the condenser section to heat it and possibly some on the flowmeter, Valve H. Valve F to the pump can be closed. The calrod heater through the EM pump should be turned on to heat the tube through the pump. This switch is located on the east wall control panel. Monitor thermocouple 57. The condenser section is heated only by hot argon flow from the immersion heater and blower; it should be watched closely to prevent a freeze-up at that section.
7. When temperatures are stabilized, the loop is ready to fill.

NOTE: If the loop is not at vacuum, it must be evacuated and outgassed per Step 13. "vacuum pulldown and outgassing," before going to the fill procedure.

## FILL PROCEDURE

Previous steps completed: Outgassing, purification system operation, weld annealing, and preheat cycle.

Control panel switches on: Master, start heat on, immersion heat, level indicator, blower.

Reference: SK-LT34, Potassium Fill System Two-Phase Loop

1. Valves open: K13, K12, K11, K9, K8, K7, K6.

Valves closed: K15, K14, K10, K5, K3, K4.

This means that the loop and fill system should be at a hard vacuum, and have outgassed during outgassing and annealing cycles. The temperatures of all points on the loop, shipper, and transfer lines should be as called out in the preheat cycle.

2. Close Valves K11, K9, K8, K7, K6, K12, K13, and 21. This should leave all valves closed.
3. The argon regulator  $R_2$  can then be set at  $1 \frac{1}{2} \pm \frac{1}{2}$  psig (make sure that upstream regulator  $R_1$  is at approximately 30 psig).
4. Open Valve K14 to allow the argon pressure to reach Valve K5 at the sump.
5. Crack Valve K4 slowly, allowing potassium to fill the manifold, and then open it fully.
6. Open K7 and fill the sample finger (you should be able to feel the potassium flow). Close K7 tightly when the flow has stopped and the sample finger is full.
7. Open K5 allowing argon pressure on potassium ( $1 \frac{1}{2}$  psig). Open K9, slowly, and allow potassium to fill the loop and surge tank to the desired level. Close K9 tightly when the level is obtained. See the instrument section for the spark probe level indicator.
8. Close Valve K5 tightly.
9. Increase pressure on the argon line with regulator  $R_2$  to 2.5 psig. It is now ready for blowback of potassium into the shipper.

10. Open K8 to pressurize the manifold and blow potassium in the manifold back into the sump.
11. Close Valves K4 and K8, in that order.
12. Open Valve K11. This pressurizes the loop and forces the liquid potassium level down in the surge tank to fill the entire loop for liquid potassium circulation. Watch temperatures closely, particularly the condenser section (Thermocouple 1) for cold spots that may freeze the potassium. Adjust the temperatures as necessary prior to starting the EM pump and circulating the potassium.

## SHAKEDOWN RUN WITH LIQUID POTASSIUM

Last Step Completed: Fill Procedure

Control panel switches on: Master, start heat on, immersion heat, level indicator, blower, Varidrive.

1. In Step 15 of the fill procedure, the loop was pressurized through the surge tank to fill the entire loop with liquid potassium.
2. Increase the loop temperature to 600°F maximum at Thermocouple 39 with main loop heaters by using the heater selection system. Watch Thermocouples 55, 56, and 57 for overheat temperatures (the maximum coil temperature allowable is 350°F). The two cold spots will undoubtedly be the flowmeter and the condenser section; however, during preheat, the blower and immersion heater have been operating and should now be running. Using Valve H, it should be possible to keep these areas from freezing potassium.

Maximum-Temperature Units:

13		9	Flowmeter 61
17	600°F	11 >200	>200°F <600°
19 Loop	Maximum	45 <600°F	Bus Bar 57
27	Range	47	>200°F <600°
31		53	
		54	Surge Tank

3. There may be some convection flow due to heat-up. It may or may not be detectable. Use the most sensitive scale on the Hewlett Packard Voltmeter (located under the meter control panel). Make sure all leads and connections are correct. Have Instrumentation personnel check this if there is any doubt. Make sure flow is in the right direction. Reverse if necessary (call Electrical). (The polarity of the readout voltmeter can be reversed by a switch on the voltmeter.)
4. When temperatures are stabilized at 600°F at Thermocouple 39, the EM pump can be energized to start liquid potassium flowing (see 5 and 6).



5. Press "PUMP INCREASE-DECREASE" switch No. 18 on decrease, to run the Variac down to the lowest limit before applying voltage to the EM pump. Press switch No. 19 to 0-150 so that voltage will be read on the 0-150 volt-range voltmeter. Turn down the low-limit settings all the way on Simplitrols 7, 8, 9, and 10, or below the reading on each control thermocouple.
6. Press "PUMP START A." This supplies voltage to the EM pump. Flow can be varied by using the increase-decrease switch No. 18 to a maximum of 25 percent power. This also drops the "START HEAT" circuits out and drops the immersion heater out of the system. The low-flow limiter (No. 22) circuit will not be operating during the time that "PUMP START A" is on, nor do the alarm panel Simplitrols have any control. Flow may be slow in getting started. It may take a few hours to get a good indication of flow due to the "wetting" action of potassium to Cb/Zr, or it may start immediately.

Monitor Thermocouples No. 55 and No. 13 for indication of overheat. Reduce power if heat is over 350°F.
7. When flow has started, and is under control and stabilized, the low-flow set point on the limiter, No. 22 on the control panel, can be adjusted to half of the measured flow. "PUMP START B" may be energized; this will put the low-flow limiter protection into operation, and it will also start the timer that sweeps the alarm circuits, putting all of the Simplitrol circuits into operation.
8. The loop is now under full protection of alarm and shutdown circuits, and temperature can be increased in preparation for the boiling cycle.
9. Increase flow to 0.25 lb per minute by adjusting increase switch No. 18. Monitor Thermocouple No. 57 closely.
10. Turn on Valve No. 30 full and control the cooling water outlet with Valve No. 31 to obtain argon discharge temperature of 200°F at Thermocouple No. 88 or No. 100. Reduce to 100°F when full flow and temperature are obtained.
11. Maintain this condition for a time designated by the E.I.C.

## BOILING OPERATION

- A. Status of loop after shakedown run with potassium.
1. Temperature =  $600^{\circ}\text{F}$  at Thermocouple 39 (low  $\Delta T$ )
  2. Flow = 0.25 lb per min
  3. Nonboiling (all liquid) = loop argon pressure set at 17.5 psia.
  4. Argon coolant flow to loop = as low as possible to maintain the isothermal condition.
  5. Argon coolant being continuously purified with purifiers set at  $800^{\circ}\text{F}$  and  $1500^{\circ}\text{F}$  (only one set of purifiers and one molecular sieve being used).
- B. Set surge tank pressure at 25 psia by adjusting  $R_2$  and flow rate at maximum available (approximately 0.98 lb per min). Monitor Thermocouple No. 57 closely.
- C. Establish heat balance in loop with Thermocouples No. 7, 13, 27, 31, and 39 steady at  $1100^{\circ} \pm 100^{\circ}\text{F}$ . Keep groups of heater elements at equal power inputs; i.e., the two preheaters should have the same power input, the three boiler elements should have the same power input, etc.
- D. While maintaining Thermocouples No. 7, 13, 27, and 31 at  $1100^{\circ}\text{F}$ , increase the superheater exit element by approximately 1.5 kw to bring the superheater exit temperature (Thermocouple No. 39) to  $1580^{\circ}\text{F}$ . During this step it will be necessary to increase argon flow to the condenser to maintain Thermocouple No. 7 at  $1100^{\circ}\text{F}$ .

At this condition a slight increase in superheater power should initiate boiling at the test-section exit.

Therefore the level monitor in the accumulator should be observed to determine when boiling starts. (Note that the level indicator will show a gradual rise while potassium in the loop is heating up in Step D. but when boiling starts there should be a greater increase in liquid level.)

- E. Initiate boiling by cautiously increasing power to the superheater exit element. (DANGER POINT) When boiling starts, the flow will drop, and quality will increase due to the negative slope of flow-vs-pressure drop in two-phase flow. An equilibrium position will be reached due to the power-input limitation, but temperatures in the boiler and preheater may be expected to rise sharply to a value

somewhere between 1600°F and 1900°F, and the flow rate should steady out at somewhere between 0.9 and 0.6 lb per minute. At this point all the boiling is taking place at the test section.

Regulate (reduce) argon pressure at the accumulator to limit temperature in the boiler and superheater to 1950°F at Thermocouple 39.

- F. After the system has stabilized at the conditions in Step E, begin increasing power slowly to the superheater exit element to shift the boiling interface from the test section to the superheater section. There are two ways to determine when the interface has shifted: (1) the flow will probably show great instability, and (2) the temperatures at Thermocouple 39 will stop rising and begin falling slowly. (DANGER POINT) If instability becomes excessive, decrease heat slightly to the superheater and allow the loop to stabilize. Then suddenly increase power to the superheater to a level at least 500 watts greater than the power level at which instability became excessive. (If this does not work, then go back to conditions preceding instability and attempt to shift boiling interface by reducing pressure at the accumulator.)

During this operation, as before, the potassium temperature from the condenser outlet to the boiler outlet should be maintained constant at 1100°F. This will require reducing preheater and boiler power and increasing argon flow to the condenser.

- G. The final step in reaching the desired operating conditions is to establish superheated vapor. This may be done by slowly increasing power to the superheater and monitoring Thermocouple 39 to determine when superheating is taking place. Note that even though Thermocouple 39 would be decreasing due to line pressure drop in the preheater as quality goes up, it (Thermocouple 39) may actually be increasing due to the general increase in  $\Delta P$  at the test section, and resulting in an increase in pressure level at the preheater. However, when superheating begins, the temperature at Thermocouple 39 will begin rising at a much greater rate. Note that all the preheating boiling and superheating is being done in the superheater section. The power required to do this is approximately 3.5 kw to heat loss, 1.5 kw to preheat, 6.9 kw to boil, and 0.079 kw to superheat (50°F).

# THERMOCOUPLE LOCATIONS

<u>T/C NO.</u>	<u>TYPE T/C</u>	<u>LOCATION</u>	<u>CONAX NO.</u>
1	PT	Condenser out	A
2	CA	Condenser out (No. 1 jct. temp.)	A
3	PT	Adiabatic condenser out	A
4	CA	Adiabatic condenser out (No. 2 jct. temp.)	A
5	PT	EM pump inlet condenser out Simplitrol No. 10	B
6	CA	EM pump inlet (No. 5 jct. temp.)	B
7	PT	EM pump inlet	B
8	CA	EM pump inlet (No. 7 jct. temp.)	B
9	PT	Adiabatic pump inlet	C
10	CA	Adiabatic pump inlet (No. 9 jct. temp.)	C
11	PT	Adiabatic pump inlet	C
12	CA	Adiabatic pump inlet (No. 11 jct. temp.)	C
13	PT	EM pump outlet	D
14	CA	EM pump outlet (No. 13 jct. temp.)	D
15	PT	Adiabatic pump outlet	D
16	CA	Adiabatic pump outlet No. 15 jct. temp.)	D
17	PT	Preheater inlet	E
18	CA	Preheater inlet (No. 17 jct temp.)	E
19	PT	Preheater inlet	E
20	CA	Preheater inlet (No. 19 jct. temp.)	E
21	PT	Adiabatic preheater inlet	F
22	CA	Adiabatic preheater inlet (No. 21 jct. temp.)	F
23	PT	Adiabatic preheater inlet	F
24	CA	Adiabatic preheater inlet (No. 23 jct. temp.)	F
25	PT	Boiler inlet (Preheater out-Simplitrol No. 7)	G
26	CA	Boiler inlet (No. 25 jct. temp.)	G
27	PT	Boiler inlet	G
28	CA	Boiler inlet (No. 27 jct. temp.)	G
29	PT	Boiler heater temp. (Heater selector sw. to Brown)	H
30	CA	Boiler heater temp. (No. 29 jct. temp.)	H
31	PT	Superheater inlet	H
32	CA	Superheater inlet (No. 31 jct. temp.)	H
33	PT	Superheater inlet (boiler out-Simplitrol No. 9)	J
34	CA	Superheater inlet (No. 33 jct. temp.)	J
35	PT	Superheater (Heater selector sw. to Brown)	J
36	CA	Superheater (No. 35 jct. temp.)	J
37	PT	Superheater outlet (Simplitrol No. 8)	K
38	CA	Superheater outlet (No. 37 jct. temp.)	K
39	PT	Superheater outlet	K
40	CA	Superheater outlet (No. 39 jct. temp.)	K
41	PT	Adiabatic superheater outlet	L
42	CA	Adiabatic superheater outlet (No. 41 jct. temp.)	L
43	PT	Adiabatic superheater outlet	L
44	CA	Adiabatic superheater outiet (No. 43 jct. temp.)	L
45	PT	Condenser inlet	M
46	CA	Condenser inlet (No. 45 jct. temp.)	M

<u>T/C</u> <u>NO.</u>	<u>TYPE</u> <u>T/C</u>	<u>LOCATION</u>	<u>CONAX</u> <u>NO</u>
47	PT	Condenser inlet	M
48	CA	Condenser inlet (No. 47 jct. temp.)	M
49	PT	Adiabatic test chamber out	N
50	CA	Adiabatic test chamber out (No. 49 jct. temp.)	N
51	PT	Adiabatic test chamber out	N
52	CA	Adiabatic test chamber out (No. 51 jct. temp.)	N
53	CA	Accumulator (surge tank thermocouple well)	P
54	CA	Accumulator skin temp. (surge tank)	P
55	CA	EM pump (bus bar)	P
56	CA	EM pump (windings)	P
57	CA	EM pump (windings)	R
58	CA	Flowmeter (magnet)	R
59	CA	Flowmeter (magnet)	R
60	CA	Condenser cooling argon	R
61	CA	Flowmeter tube temp.	S
62	CU	Flowmeter MV signal	S
63	CA	Flowmeter cooling argon	S
64	CA	Argon return temp.	S
65	CA	EM pump cooling argon	T
66	CA	Expansion loop, Cb/Zr Additional thermocouples will be assigned for shipper transfer lines, and manifold valves, sample fingers, etc.	
67	CA	Sampling tube, preheat sect.	
68	CA	Ambient temp. (on channel stiffener)	
69		Top center of the chamber door mounting flange	
70		Immersion heater discharge	
71		Cooling blower discharge	
72		Cooling blower inlet	
73		Potassium sump tank, skin	
74		Valve K4	
75		Sample finger (dump)	
76		Fill manifold (dump sample finger)	
77		Fill manifold (tee, argon inlet line)	
78		Fill manifold (chamber inlet)	
79		Sample finger (fill)	
80		Fill manifold (fill sample finger)	
81		Transfer line, inlet to sump	
82		Potassium sump tank, skin	
83		Transfer line, shipper discharge	
84		Shipper valve K-1	
85		Shipper, top	
86		Shipper, center	
87		Shipper, bottom	
88		Argon - H <sub>2</sub> O Hx - gas discharge	
89		Argon - H <sub>2</sub> O Hx - gas inlet	
90		Bottom center of chamber door mount flange	
91		Top purifier 1-1	
92		Bottom purifier 1-1	
93		Top purifier 1-2	

<u>T/C</u> <u>NO.</u>	<u>TYPE</u> <u>T/C</u>	<u>LOCATION</u>	<u>CONAX</u> <u>NO.</u>
94		Bottom purifier 1-2	
95		Top purifier 2-3	
96		Bottom purifier 2-3	
97		Top purifier 2-4	
98		Bottom purifier 2-4	
99		Well on gettering sump	
100		Argon - H <sub>2</sub> O Hx - gas discharge	
101		Purification blower discharge (blower box)	
102		Immersion heater discharge	
103		Purified argon inlet to chamber	
104		Purified argon inlet to chamber	
105		Purified argon inlet to sampling manifold	
106		Sampling manifold	
107		Sampling manifold	
108		Bank 2 argon purifier discharge	
109		Bank 1 argon purifier discharge	
110		Argon inlet to surge tank	
111		Bubbler system temp.	
112		Inst. port flange (blank off plate)	
113		Thermocouple port flange	
114		Power port flange	
115		Power port flange	
116		Thermocouple port flange	
117		Circulating blower motor	
118		Circulating blower bearing	
119		Purification blower bearing, inlet side	
120		Purification blower motor	
121		Purification blower bearing, discharge side	
122		Scanner oil bath	
123		Molecular sieve, top	
124		Inlet to high-temp. furnace for 1-1	
125		Discharge of high-temp. furnace for 1-1	
126		Bottom element temp. for 1-1	
127		Inlet to high-temp. furnace for 2-3	
128		Discharge of high-temp. furnace for 2-3	
129		Bottom element temp. for 2-3	
130		Skin temp. under clam shells, 1-2	
131		Skin temp. under clam shells, 1-2	
132		Skin temp. under clam shells, 2-4	
133		Skin temp. under clam shells, 2-4	